



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of  
SANFORD M. STEVENSON  
Serial No. 09/652,272  
Filed: August 31, 2000  
FOR: METHOD FOR REMOVING METAL  
COMPOUNDS FROM WASTE WATER

) Art Unit 1724

) Examiner: P. Hruska

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PETITION TO INSTITUTE PUBLIC USE PROCEEDING  
PURSUANT TO 37 C.F.R. § 1.292

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July 31, 2001

Petitioner

5/07



# 11

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of )  
SANFORD M. STEVENSON )  
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Commissioner for Patents  
Washington, D.C. 20231

**PUBLIC PROTEST PURSUANT TO 37 C.F.R. § 1.291**

Pursuant to 37 C.F.R. § 1.291, the undersigned protests pending U.S. Patent Application Serial No. 09/652,272 (the "Application"), on the basis that U.S. Patent No. 4,652,381 ("Inglis") (Tab A), issued March 24, 1987, has not been considered in its prosecution and that the applicant has not properly fulfilled the duty of candor required by 37 C.F.R. § 1.56.

**SERVICE**

In accordance with 37 C.F.R. § 1.248, this protest and its attachments were served on Louis M. Tarasi, Jr., litigation attorney for the applicant, and Daniel J. Long, prosecuting attorney for the applicant, on July 31, 2001.

**BACKGROUND**

The Application was filed on August 31, 2000, claiming priority from U.S. Patent Application Serial No. 08/072,412, filed May 25, 1993, now U.S. Patent No. 5,370,800 (the "'800 patent"). Applicant sued the United States in January 1997 pursuant to

28 U.S.C. § 1498, and the undersigned is defense counsel in that litigation. See Chemical Separation Technology, Inc., et al. v. United States, 45 Fed.Cl. 513 (1999). On November 14, 2000, pursuant to Markman v. Westview Instruments, Inc., 517 U.S. 300 (1996), the Court issued an oral ruling construing the claims of the '800 patent. Transcript (Tab B). Trial to the Court on invalidity was held November 15-17, 2000, but a decision has not yet been issued.

On July 20, 2001, the applicant notified the Court and the undersigned that the Application's Notice of Allowance had been mailed. July 20 Letter (Tab C). The applicant provided a copy of the Application's prosecution history to the Court and to the undersigned on July 24, 2001. July 24 Letter (Tab D). Until July 20, 2001, the undersigned was unaware of the pending Application.

#### DISCUSSION

##### I. Inglis Renders the Pending Claims Unpatentable

The Application contains 25 allowed claims, all of which depend from claim 7. Claim 7 recites a method of removing copper from waste water, comprising simultaneous pH adjustment, aeration, and agitation in a reaction tank, followed by polymer addition, flocculation, and separation. Claim 7 of the Application is substantively identical to claim 1 of the '800 patent, except for its limitation to copper, one member of the eight-member Markush group recited in the preamble to claim 1 of the '800 patent.

Dependent claims 8 through 31 are otherwise identical to dependent claims 2 through 25, respectively, of the '800 patent.

Inglis issued on March 24, 1987, making it prior art to the Application under 35 U.S.C. § 102(b). Inglis (Tab A). Like the Application, Inglis claims a method of removing copper (among other metals) from waste water, comprising simultaneous pH adjustment, aeration, and agitation in a reaction tank, followed by polymer addition, flocculation, and separation. In fact, Inglis differs from Application claim 7 only in that Inglis' copper concentration, waste water flow rate, and "dissolved oxygen concentration" are lower than that recited in claim 7:

<b>Application Claim 7</b>	<b>U.S. Patent 4,652,381 (Inglis)</b>
A method for removing metal compounds comprising copper metal compounds from waste water comprising the steps of:	Copper is removed as a carbonate salt. Col. 3, ll. 47-57.
(a) adjusting the pH of the waste water to from about 5 to about 12;	"pH of the waste water contained in the treatment tank 28 [is] maintained at a pH of from about 4.8 to 5." Col. 6, ll. 39-41.  pH of wastewater leaving reactor is "in the order of 5 to 7" Col. 7, l. 12.
(b) aerating the waste water;	"Air is sparged continuously into the interior of treatment tank 28" Col. 6, ll. 11-12.

<p>(c) agitating the waste water, where steps (a), (b), and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a copper metal concentration of from about 50 mg/l. to about 1,000 mg/l.</p>	<p>"Air is sparged continuously into the interior of treatment tank 28 through line 38 to effect some mixing of the liquid" Col. 6, ll. 11-13.</p> <p>"the present method treats 1,200 gallons a day" Col. 3, ll. 64-65.</p> <p>"copper can be as high as 10 ppm." Col. 5, ll. 43-44.</p>
<p>(d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said copper metal compounds to form; and</p>	<p>"an anionic polyelectrolyte flocculant contained in tank 92 is pumped via line 94 into the final treatment tank 50." Col. 7, ll. 58-60.</p>
<p>(e) then separating said floccules including said copper metal compounds from the water.</p>	<p>"the contents of final treatment tank 50 are allowed to settle" Col. 7, ll. 64-65.</p>

Nothing in Inglis suggests that a higher flow cannot be processed, or that the specific operating parameters are significant. Inglis' preferred embodiment employs batch processing, but it may also be used continuously. Col. 5, l. 54. Since the copper concentration and waste water flow rate depend on conditions of use, higher operating ranges - for example, "about 50 gal./min." flow rate and "about 50 mg/l." metal concentration - are inherent. Moreover, the "dissolved oxygen concentration" is inherently adjustable and dependent, in part, on the concentration of oxidizable metals in the waste water. Col. 6, ll. 14-16.

Indeed, that Inglis' claims say nothing about operating parameters strongly suggests that those parameters are not crucial. Inglis therefore anticipates claim 7 of the Application.

This conclusion is bolstered by the Court's construction of the claims of the '800 patent. Specifically, the Court concluded that the listed operating ranges "are not intended as limitations on the patent." Transcript at 414:24-25 (Tab B). That is, any flow rate and any metal concentration satisfy the requirements of claim 1 of the '800 patent, and the same must also be true of claim 7 of the Application. Thus construed, the sole difference between Inglis and claim 7 is no difference at all, and claim 7 is anticipated.

Dependent claims 8, 12, 27, and 28 are similarly anticipated by Inglis: belt filter 64 performs the "further dewatering" of claim 8, treatment tank 50 functions as the clarifier of claim 12, and anionic polymer is added to treatment tank 50, which performs both the clarification required by claim 27 and the settling required by claim 28. Finally, the other dependent claims of the Application are obvious variations of claim 7, since they simply recite different configurations of equipment well known in the art. This was the examiner's conclusion during prosecution of the '800 patent, when all claims were initially rejected as obvious over various combinations of prior art water treatment systems. '800 File at D024746-50 (Tab E). The same is true of the present

Application, and dependent claims 8-31 thus fall with claim 7.

II. The Applicant has not Properly Satisfied his Duty of Candor

On May 14, 2001, six days after reaching agreement with the examiner regarding the claim language, and three days before the Notice of Allowance issued, the Applicant filed a Supplemental Information Disclosure Statement. The Statement cites three prior art references: Inglis, the "EPA Design Manual Neutralization of Acid Mine Drainage," and a pre-critical date "request for a quote based on drawings." May 14 Information Disclosure Statement. All three references were known to the Applicant before the filing date of the Application, since they are the subject of litigation in Chemical Separation Technology v. United States. The "request for a quote" creates an on sale bar, as discussed in the co-pending Petition for Public Use Proceeding, and was the principal issue in Chemical Separation Technology, 45 Fed.Cl. 513, where the Court held that a pre-critical date, non-experimental offer for sale of an embodiment of the claims of the '800 patent had been made. The EPA Design Manual was discussed at length in the June 22, 2000 report of John A. Roth, the expert witness who testified at trial on behalf of the Applicant. Roth Expert Report at 6-11 (Tab F). Inglis was discussed in detail in the Supplemental Declaration of Daniel Dupon (expert witness for the defense), which was completed June 30, 2000 and delivered to the Applicant on July 7, 2000. Dupon Expert Report and Cover Letter (Tab G). The Information

Disclosure Statement fails to mention the Court of Federal Claims' November 14, 2000 construction of virtually identical claim language in the '800 patent. Transcript (Tab B).

37 C.F.R. § 1.56 requires inventors to disclose "all information known to that individual to be material to patentability." The three prior art references were not disclosed until after reaching agreement with the examiner and, thus, after prosecution was effectively complete, even though all three were known to the inventor long before the filing of the Application. They are clearly highly material; in fact, the Applicant has attempted to offer the Application's Notice of Allowance to the Court as evidence of the validity of the '800 patent over all three references. July 20 Letter (Tab C).

Waiting until prosecution is complete before submitting highly material prior art does not constitute a proper discharge of the Applicant's duty of candor. Just as "'burying' a particularly material reference in a prior art statement containing a multiplicity of other references can be probative of bad faith," delaying disclosure until it is effectively too late for the examiner to consider it evidences an intent to subvert the examination. Molins, PLC v. Textron, Inc., 48 F.3d 1172, 1184 (Fed. Cir. 1995). Similarly, the failure to disclose the Court's construction of the virtually identical claims of the '800 patent demonstrates the Applicant's disregard of his duty of candor and

lack of respect for both the Court and the examiner. All claims of the Application should be rejected for violation of that duty, in accordance with 37 C.F.R. § 1.56(a).

Respectfully submitted,

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Director

July 31, 2001

  
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Attachments: Petitioner

- A) U.S. Patent No. 4,652,381
- B) Markman hearing transcript
- C) July 20 Letter
- D) July 24 Letter
- E) File wrapper excerpts,  
U.S. Patent No. 5,370,800
- F) Roth expert report
- G) Dupon expert report



THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of )  
SANFORD M. STEVENSON ) Art Unit 1724  
Serial No. 09/652,272 ) Examiner: P. Hruskoci  
Filed: August 31, 2000 )  
FOR: METHOD FOR REMOVING METAL )  
COMPOUNDS FROM WASTE WATER )

Commissioner for Patents  
Washington, D.C. 20231

**PETITION TO INSTITUTE PUBLIC USE PROCEEDING  
PURSUANT TO 37 C.F.R. § 1.292**

Pursuant to 37 C.F.R. § 1.292, the undersigned petitions to institute a public use proceeding regarding pending U.S. Patent Application Serial No. 09/652,272 (the "Application"), on the basis that its claims are anticipated or rendered obvious by an offer for sale and a public use more than one year prior to May 25, 1993.

**SERVICE**

In accordance with 37 C.F.R. § 1.248, this petition and its attachments were served on Louis M. Tarasi, Jr., litigation attorney for the applicant, and Daniel J. Long, prosecuting attorney for the applicant, on July 31, 2001.

**BACKGROUND**

The Application was filed on August 31, 2000, claiming priority from U.S. Patent Application Serial No. 08/072,412, filed May 25, 1993, now the '800 patent. Applicant sued the United States in January 1997 pursuant to 28 U.S.C. § 1498, and the undersigned is defense counsel in that litigation. See Chemical

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Separation Technology, Inc., et al. v. United States, 45 Fed.Cl. 513 (1999) (Tab D). Trial to the Court was held November 15-17, 2000, but a decision has not yet been issued.

No later than January 1991, the applicant operated a commercial water treatment plant near Indiana, Pennsylvania, which the parties in litigation have called the "Keystone Plant." Review and Documentation of the CST Industrial Wastewater Treatment System ("Keystone Report") (Tab A). In April 1992 the applicant offered for sale the "Portable Interim Treatment System" ("PITS"), a water treatment plant designed to remove copper from waste water. April 16, 1992 Letter (Tab B). It has been plaintiff's position that the PITS was within the scope of all 25 claims of U.S. Patent No. 5,370,800 (the "'800 patent"), the parent of the present Application. Affidavit at ¶ 3 (Tab C).

#### DISCUSSION

The Application contains 25 allowed claims, all of which depend from claim 7. Claim 7 recites a method of removing copper from waste water, comprising simultaneous pH adjustment, aeration, and agitation in a reaction tank, followed by polymer addition, flocculation, and separation. Claim 7 of the Application is substantively identical to claim 1 of the '800 patent, except for its limitation to copper, one member of the eight-member Markush group recited in the preamble to claim 1 of the '800 patent. Dependent claims 8 through 31 are otherwise identical to dependent

claims 2 through 25, respectively, of the '800 patent.

A public use proceeding is justified if the petitioner makes a prima facie showing that the claimed invention was in public use or on sale before the critical date, that is, more than one year before the filing of the application from which priority is claimed. 37 C.F.R. § 1.292(a). Two activities warrant a public use proceeding here: the commercial offer to sell the PITS, and the public use of the Keystone Plant.

I. The PITS Offer Creates an On Sale Bar

In deciding a motion for summary judgment of invalidity of the '800 patent, the Court of Federal Claims ruled that the PITS was offered for sale before May 25, 1992, and that the experimental use exception did not apply. Chemical Separation Technology, 45 Fed.Cl. at 517-18 (Tab D). This conclusion was based in part on correspondence between the applicant and his customer, documenting the design of the PITS and the expected results. April 16, 1992 Letter (Tab B); May 15 Letter (Tab E); May 15 Letter (Tab F). The only issue unresolved by the Court's summary judgment opinion was the date the claims of the '800 patent were ready for patenting. 45 Fed.Cl. at 518.

The present Application is identical to the '800 patent, except that the claims are limited to removal of copper, one of the metals the PITS was designed to treat. May 15, 1992 Letter (Tab E); April 16, 1992 Letter (Tab B). Thus, the '800 patent's on sale

bar analysis applies with equal force to the Application's on sale bar analysis: an embodiment of the claims was offered for sale before the critical date (May 15, 1992 Letter (Tab E)), according to the applicant the PITS fell within the scope of all 25 claims (Affidavit at ¶ 3 (Tab C)), and the sale was not for an experimental purpose (45 Fed.Cl. at 518). The only issue is the date the claims were ready for patenting, that is, the date they had been reduced to practice or that there existed "drawings or other descriptions of the invention that were sufficiently specific to enable a person skilled in the art to practice the invention."

Chemical Separation Technology, 45 Fed.Cl. at 517.

The applicant asserts that the claims were ready for patenting only after the invention had been "tried on non-ferrous metals," which allegedly did not occur until after the critical date. January 31, 2000 Information Disclosure Statement. Four pieces of evidence contradict this assertion, and prove that the claims were ready for patenting well before May 25, 1992. First, before the critical date the Keystone Plant had successfully removed non-ferrous metals - specifically, manganese and aluminum. Keystone Report at 8 (Tab A). It also otherwise fell within the scope of at least claim 7, as the claim chart demonstrates:

Application Claim 7	Keystone Report
A method for removing metal compounds comprising copper metal compounds from waste water comprising the steps of:	Manganese and aluminum are removed from the leachate. p. 8, Table 1.

<p>(a) adjusting the pH of the waste water to from about 5 to about 12;</p>	<p>Plant effluent has a pH of 7.1. p. 8, Table 1.</p>
<p>(b) aerating the waste water;</p>	<p>"The CST system . . . brings oxygen, alkali, and leachate together in a way that results in a fast N-O-P (neutralization-oxidation-precipitation) reaction." p. 19.</p>
<p>(c) agitating the waste water, where steps (a), (b), and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a copper metal concentration of from about 50 mg/l. to about 1,000 mg/l.</p>	<p>"The CST system achieves rapid acid neutralization and metal precipitation of coal refuse leachates by reacting the leachate with sodium hydroxide and air-derived oxygen in a patented reactor at atmospheric pressure." p. 1.</p> <p><math>3 \times 7.5 = 22.5</math> lb./hr. oxygen. p. 8, Table 1.</p> <p>230 gpm waste water flow. p. 8, Table 1.</p> <p>17 (manganese) + 114 (aluminum) = 131 mg/l non-ferrous metals concentration. p. 8, Table 1.</p>
<p>(d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said copper metal compounds to form; and</p>	<p>"To effect solids concentration and recovery, flocculants are used to bind and hold the particles together while their mass undergoes dewatering." p. 3.</p> <p>"The flocculant used at Keystone was Percol 730 (Allied Colloid)." p. 3.</p>
<p>(e) then separating said floccules including said copper metal compounds from the water.</p>	<p>"To effect solids concentration and recovery, flocculants are used to bind and hold the particles together while their mass undergoes dewatering." p. 3.</p>

Plainly, the Keystone Plant removed non-ferrous metals using a process otherwise within the scope of claim 7, and the applicant's claim that before the critical date "it was not scientifically predictable that the invention would remove non-ferrous metals" is not credible. January 31, 2000 Invention Disclosure Statement.

Second, the July 1991 Business Plan of Chemical Separation Technology, the company of which the applicant was Vice President in 1991 and is currently President, explicitly states that the claimed process will remove copper. Affidavit at ¶ 1 (Tab C); 1991 Business Plan at 13 (Tab G). In particular, the process described is capable of removing "copper, zinc, lead, and magnesium" (1991 Business Plan at 17 (Tab G)), it is essentially the same as that documented in the Keystone Report (id. at 15-17), and it is shown graphically in a figure virtually identical to figure 3 of the '800 patent (id. at 16).

Third, the PITS correspondence corroborates the applicant's successful pre-critical date removal of copper. His April 16, 1992 letter predicts an effluent copper concentration of less than 0.1 mg/l, given historical metals discharge concentrations "in the .002 Mg/l ranges." (Tab B). His May 15, 1992 letter states that mercury, lead, and cadmium will "experience some co-precipitation with Fe and Cu." (Tab F). Clearly, the applicant had sufficient experience with copper removal to be able to reduce at least claim 7 to practice, and enough confidence in the claimed invention to

sell a commercial embodiment of it by touting that experience.

Fourth, the claimed copper removal process was known in the prior art - in particular, in U.S. Patent No. 4,652,381 ("Inglis") (Tab H). As discussed in greater detail in petitioner's pending Public Protest, Inglis describes removal of copper, as well as lead and zinc, two other non-ferrous metals, using claim 7's process:

<b>Application Claim 7</b>	<b>U.S. Patent 4,652,381 (Inglis)</b>
A method for removing metal compounds comprising copper metal compounds from waste water comprising the steps of:	Copper, lead, and zinc are removed as carbonate salts. Col. 3, ll. 47-57.
(a) adjusting the pH of the waste water to from about 5 to about 12;	"pH of the waste water contained in the treatment tank 28 [is] maintained at a pH of from about 4.8 to 5." Col. 6, ll. 39-41.  pH of wastewater leaving reactor is "in the order of 5 to 7" Col. 7, l. 12.
(b) aerating the waste water;	"Air is sparged continuously into the interior of treatment tank 28" Col. 6, ll. 11-12.
(c) agitating the waste water, where steps (a), (b), and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a copper metal concentration of from about 50 mg/l. to about 1,000 mg/l.	"Air is sparged continuously into the interior of treatment tank 28 through line 38 to effect some mixing of the liquid" Col. 6, ll. 11-13.  "the present method treats 1,200 gallons a day" Col. 3, ll. 64-65.  "copper can be as high as 10 ppm." Col. 5, ll. 43-44.

(d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said copper metal compounds to form; and	"an anionic polyelectrolyte flocculant contained in tank 92 is pumped via line 94 into the final treatment tank 50." Col. 7, ll. 58-60.
(e) then separating said floccules including said copper metal compounds from the water.	"the contents of final treatment tank 50 are allowed to settle" Col. 7, ll. 64-65.

In summary, the Keystone Report demonstrates a reduction to practice of at least claim 7 on non-ferrous metals in general, the 1991 Business Plan and PITS correspondence prove the efficacy of the claimed process on copper in particular, and Inglis shows that the claims were so ready for patenting that someone else had already patented the invention, all before May 25, 1992. Since the Court has declared that the PITS was commercially offered for sale before the critical date, and since the applicant himself asserts that the PITS was an embodiment of all claims of the parent patent, a public use proceeding is justified.

## II. Operation of the Keystone Plant Creates a Public Use Bar

"Public use" includes secret commercial use by the inventor. Woodland Trust v. Flowertree Nursery, Inc., 148 F.3d 1368, 1370 (Fed. Cir. 1998). Since the applicant reduced claim 7 to practice with non-ferrous metals at the Keystone Plant, its continued use after the Keystone Report was written could not have been experimental and must have been commercial. Zacharin v. United States, 213 F.3d 1366, 1369 (Fed. Cir. 2000); 1991 Business Plant

at 2 (Tab G) (Keystone Plant was in "commercial use"). As a result, whether or not the applicant's use of the Keystone Plant after January 1991 was secret, it constitutes a barring public use if copper was removed. The 1991 Business Plan and the PITS letters memorialize the successful removal of copper. April 16, 1992 Letter (Tab B); May 15, 1992 Letter (Tab F); 1991 Business Plan at 17 (Tab G). Thus, operation of the Keystone Plant amounts to a barring public use, and a public use proceeding is warranted.

Respectfully submitted,

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Attachments: Petitioner

- A) Keystone Report
- B) April 16, 1992 Letter
- C) Stevenson Affidavit
- D) Opinion, 45 Fed.Cl. 513 (1999)
- E) May 15, 1992 Letter
- F) May 15, 1992 Letter
- G) 1991 CST Business Plan excerpt
- H) U.S. Patent No. 4,652,381

# United States Patent [19]

Inglis

[11] Patent Number: 4,652,381  
[45] Date of Patent: Mar. 24, 1987

[54] BATTERY PLANT WASTE WATER TREATMENT PROCESS

[75] Inventor: James L. Inglis, Liberty, Mo.

[73] Assignee: Farmland Industries, Inc., Kansas City, Mo.

[21] Appl. No.: 757,351

[22] Filed: Jul. 22, 1985

[51] Int. Cl. 4 C02F 1/52

[52] U.S. Cl. 210/724; 210/722;

210/726; 210/912; 423/35; 423/92

[58] Field of Search 210/712, 713, 721, 722, 210/724, 726, 727, 912; 423/92, 35

[36] References Cited

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3,617,559	11/1971	Cywin	210/722
3,931,007	1/1976	Sugano et al	210/721
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4,336,236	6/1982	Kobakowski et al	423/92
4,338,200	7/1982	Zeijlstra	423/92
4,341,636	7/1982	Harder et al	210/669

FOREIGN PATENT DOCUMENTS

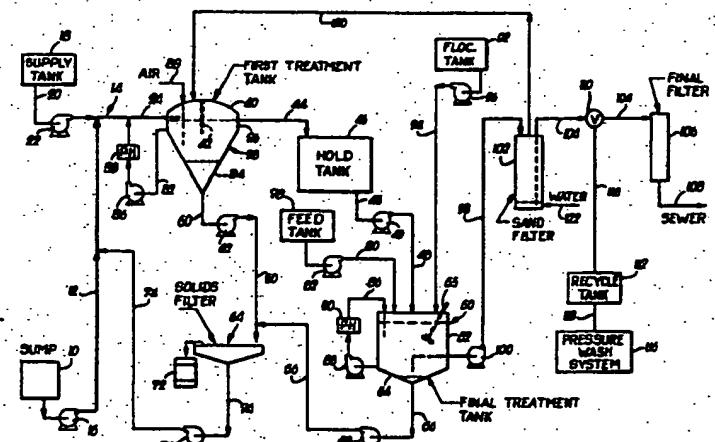
50-79169 6/1975 Japan 210/912

Primary Examiner—Peter Hruskoci  
Attorney, Agent, or Firm—Schmidt, Johnson, Hovey, & Williams

[57] ABSTRACT

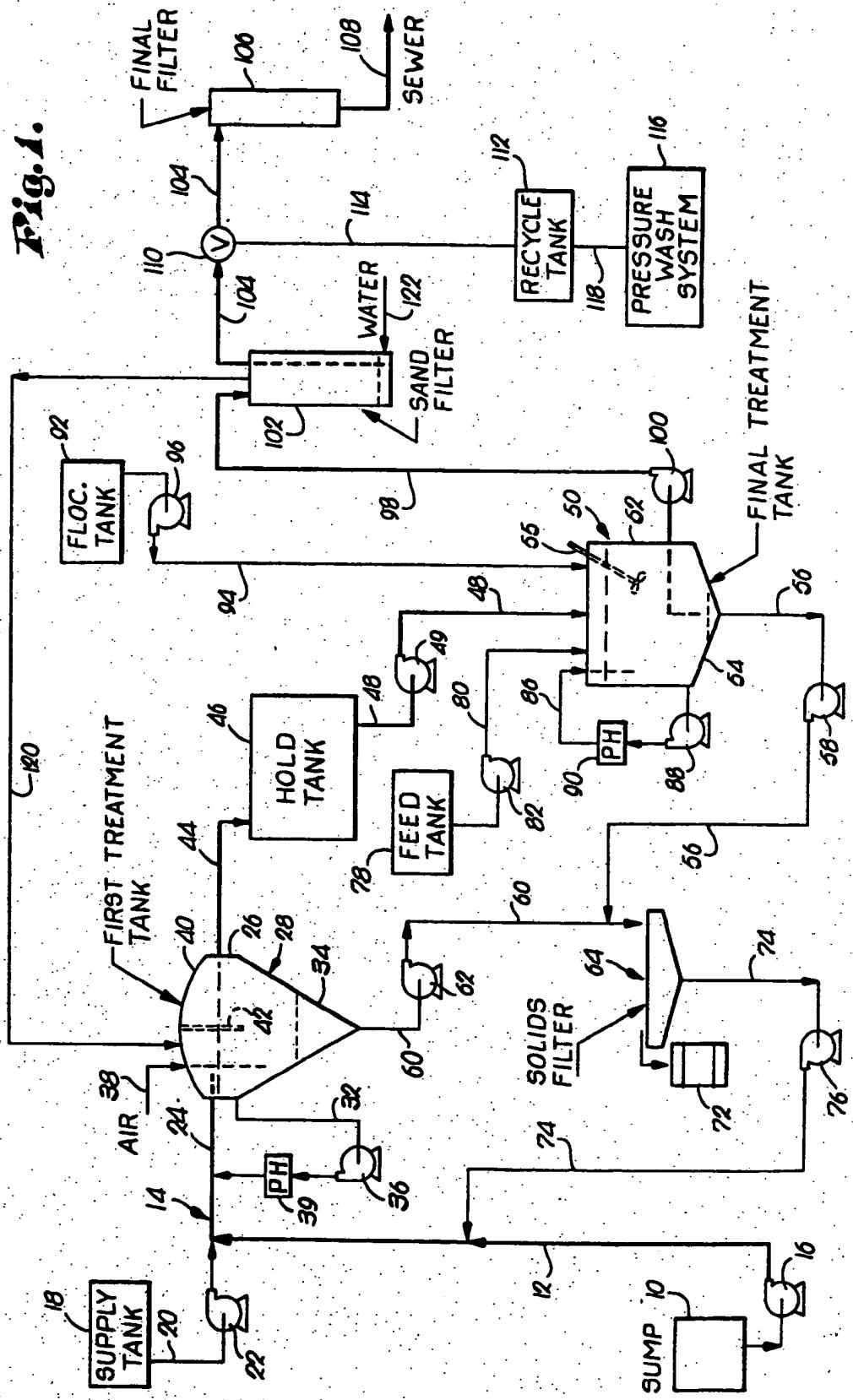
A process of treating industrial waste water contaminated with environmentally unacceptable amounts of sulfuric acid and heavy metals such as lead, copper or zinc is disclosed which permits lowering of the concentration of the contaminants to a level permitting discharge to the sewer. Waste water resulting from floor wash and spray washing of lead acid batteries prior to shipment from the manufacturing facility contains sufficient sulfuric acid to cause the pH to normally be at a level of about 2 along with heavy metal contaminants present in concentrations which require treatment for removal before the discharge water will meet EPA standards. The water to be treated is directed to a first reaction and settling vessel where calcium carbonate is added along with an oxidation medium such as air which also functions to stir the stored waste water. Sufficient calcium carbonate is added to bring the pH of the solution to a level of about 5 and at the same time react with the heavy metals present such as lead, copper or zinc. Calcium sulfate and respective heavy metal carbonates precipitate and settle to the bottom of the treatment zone where they may be readily removed. In a second treatment vessel, calcium hydroxide along with enough calcium carbonate to maintain an excess of carbonate ion are added to complete separation of the heavy metals. Final removal of precipitate from the solution is accomplished through a suitable filter.

3 Claims, 7 Drawing Figures



D024956

## Fig. 1.



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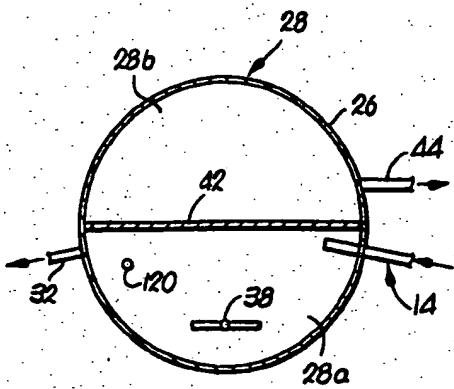


Fig. 3.

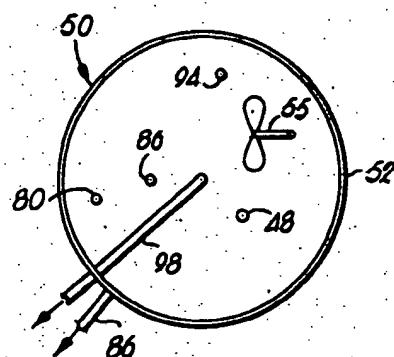


Fig. 5.

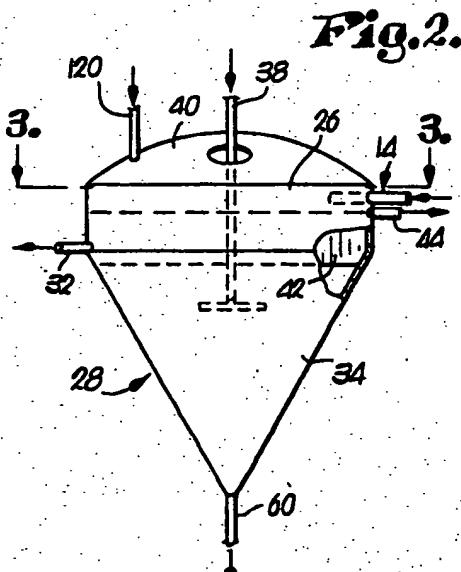


Fig. 2.

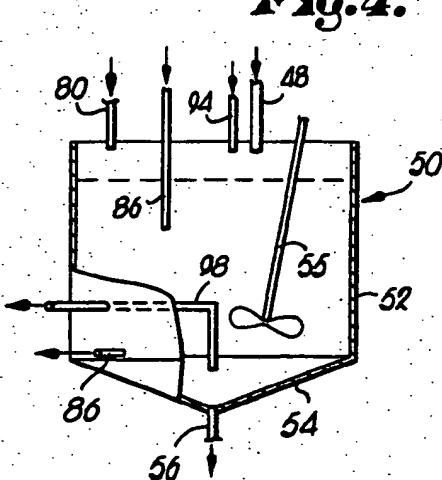


Fig. 4.

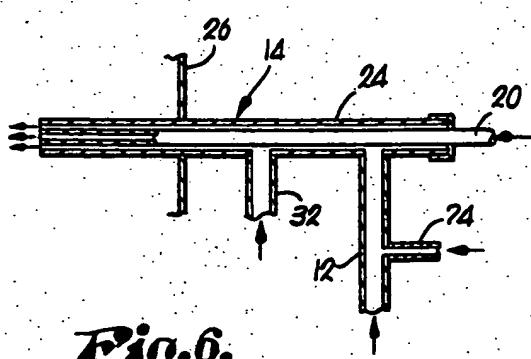


Fig. 6.

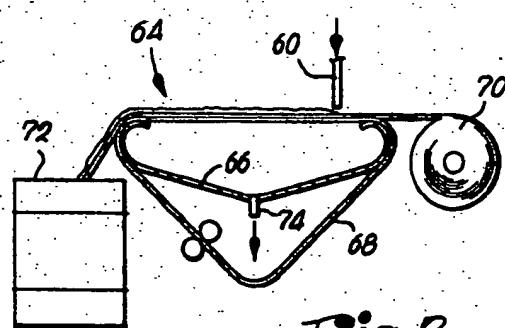


Fig. 7.

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BATTERY PLANT WASTE WATER TREATMENT  
PROCESS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a process for treating industrial waste water containing contaminants in the nature of sulfuric acid and heavy metals such as lead, copper and zinc, to lower the concentration of such contaminants to a level permitting discharge of the waste water to the sewer.

The process has particular utility in the treatment of waste water resulting from the washing of lead acid batteries and plant floors causing the water to contain residual quantities of sulfuric acid and metal constituents primarily made up of lead and copper.

Use of the process permits recycling of treated water back to the plant washing process thus lowering the overall operating costs of the battery plant. Water which is periodically discharged to the sewer has acid and metal levels far below those permitted in governmental regulations.

2. Description of the Prior Art

The assignee of this application has long operated a lead acid battery plant. During the manufacture of lead acid storage batteries, it has been conventional for some time to suspend the batteries from a conveyor line for passage through a water spray cleaning system. Water is sprayed onto the batteries to thoroughly clean the exterior surface of each battery. The spent water is collected through floor drains and directed to a storage area. Following washing of the battery cases, the batteries are generally dried with pressurized air. A large amount of waste water also comes from washing down the floor in the plant.

The waste water from the lead acid battery wash and plant floor washings cannot be reused in the manufacturing process in part because iron in the waste water is deleterious to battery life, and the high acidity and lead content preclude discharge direct to a sewer without subjecting the water to a treatment operation. In the past, the assignee hereof has employed a mixture of sodium hydroxide and sodium sulfide to remove lead from the waste water. Some recycling of the contaminated water was carried out, but build up of a sodium sulfate salt prevented effective use of the treatment on an ongoing basis. EPA regulations provide that only 0.15 lb. of lead and 0.76 lb. of copper may be found in 50 lb. of waste water per million pounds of lead used per month in order to permit discharge of the waste water into the sewer.

It has been suggested in "Development Document for Effluent Limitation Guidelines and Standards for the Battery Manufacturing Point Source Category", October 1982, EPA 440/1-82/067-b, U.S. Environmental Protection Agency, that sodium hydroxide be added to the waste water to be treated and the resulting  $Pb(OH)_2$  allowed to settle for removal.  $Pb(OH)_2$  has a minimum solubility point on its solubility curve at pH 9.5. Thus, the discharge to sewer should be 9 to 10 and never below 8. Below pH 8, the solubility of  $Pb(OH)_2$  is such that meeting the discharge requirements would be very difficult.

The development document also describes a method of using soda ash ( $Na_2CO_3$ ) and caustic soda ( $NaOH$ ) to treat the waste when no recycling is required to give

low lead concentration. In this procedure, the lead is precipitated as the carbonate at a pH above 7.

Hautala, et al. in Volume 11, Issue No. 1 at pages 243, et seq. (1977) of Water Research ("Calcium Carbonate in the Removal of Iron and Lead From Dilute Waste Water") indicate that calcium carbonate has been used in the treatment of acid mine waters and steel pickle liquors at pH levels of 8 or more. However, precipitation of ferric hydroxide is believed to interfere with settling of calcium sulfate and lead carbonate. The Hautala, et al. process does not provide a treated waste water which will satisfy current EPA sewer discharge regulations.

Hoak, et al. in Volume 37, Issue No. 6, Industrial and Engineering Chemistry, June 1945 at pages 553, et seq. ("Treatment of Spent Pickling Liquors with Limestone and Lime") describe a process of treating spent pickling liquor with limestone and lime. Quick lime ( $CaO$ ) was used to elevate the pH to the alkaline side before air was added to achieve rapid oxidation.

SUMMARY OF THE INVENTION

Waste water resulting from washing the exterior surfaces of lead acid batteries and from washing the plant floor is stored and when a suitable quantity thereof is collected, it is directed into a combination reaction and settling vessel wherein calcium carbonate is metered into the waste water to maintain a pH of about 5. Air is introduced into the waste water in the reaction vessel for the dual purpose of agitating the solution and assuring intimate admixture of the calcium carbonate and water solution, and to oxidize iron present in the waste water to ferric oxide precipitate. Calcium sulfate and lead, copper, zinc and other heavy metal carbonates thus formed settle out upon standing of the solution and may be removed from the treatment vessel.

The liquid is directed to a suitable hold tank and then introduced into a final treatment vessel where the solution is mechanically agitated and a slurry of about 12% by weight of mixed limes (20% calcium carbonate and 80% calcium hydroxide) is added. Again, precipitates are allowed to settle out with the clarified solution being directed overhead and then passed through a sand filter to effect final removal of a remaining solids from the water, which is then in condition for recycling to the plant wash water system.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic diagram of a preferred process for carrying out the invention hereof and illustrates in diagrammatic form various pieces of equipment which may be advantageously employed to practice the process;

FIG. 2 is an enlarged fragmentary view of the first treatment tank on a somewhat enlarged scale illustrating the construction of a preferred vessel and the manner in which liquid and gas are caused to inflow and liquid is allowed to outflow therefrom;

FIG. 3 is a horizontal cross-sectional view taken substantially along the line 3-3 of FIG. 2;

FIG. 4 is an enlarged fragmentary view of the final treatment tank again illustrating the construction of a preferred vessel and the manner in which liquid is caused to inflow and outflow therefrom;

FIG. 5 is a horizontal view looking downwardly on the open top vessel of FIG. 4;

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FIG. 6 is a depiction of suitable flow direction piping for the liquid streams introduced into the vessel of FIG. 2; and

FIG. 7 is a schematic illustration of the solids filter used in association with the final treatment tank of FIG. 4.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

In a typical lead acid battery plant which has a total lead input of approximately 1,000,000 pounds per month, current EPA regulations limit the waste water discharge to sewer contaminants to 0.15 lb. of lead and 0.76 lb. copper per month. If 50,000 lbs. of lead per day is consumed, the daily limit of lead and copper in waste water to sewer would be no more than 0.016 lb. of lead and 0.072 lb. of copper. Current EPA regulations are generally based on the total amount of contaminants discharged over a selected period of time and not their concentration in the waste stream. Compliance is determined by measuring the concentrations of the metals in the waste stream and multiplying it by the waste stream volume. New regulations thereby encourage the reduction of waste stream volumes. With a lead concentration of 1.28 ppm in the waste stream, only 1,200 gallons of waste water could be discharged in a 24 hour period. However, at 0.2 ppm of lead in the waste water, 7,600 gallons of treated waste water could be discharged to the sewer in a 24 hour period.

The process of this invention produces a waste water discharge to sewer containing less than 0.2 ppm of lead and an even lesser amount of copper. This compares with prior processes which gave a soluble lead concentration in the waste water above 10 ppm and to a certain extent were erratic and unpredictable.

Metal contaminants are precipitated as carbonate and carbonate-hydroxide basic salts. The solids are separated by filtration and sent to a landfill. It is a feature of this process that precipitation of these salts is affected at a pH value lower than those indicated to be feasible in the prior art. Precipitation of lead, copper and zinc carbonate salts have been found to occur at a pH as low as 4.5 to 5.0. The carbonates thus precipitated at a pH below 7 are less soluble than lead, copper and zinc hydroxide which are precipitated at a pH above 7. As a result, the removal of lead, copper and zinc salts is more efficient using a carbonate system than is the case when reliance is placed on precipitation of the metals as hydroxides above pH 7, as in the processes of the prior art and particularly, the EPA referenced methods. It is also a feature of this process that trace amounts of iron are removed by sparging air into the treatment vessel to precipitate the iron as  $Fe_2O_3$ . Precipitation of the iron by oxidation with air may readily be accomplished without adversely affecting removal of lead, copper and zinc as carbonates in an acidic medium. Furthermore, removal of the iron as ferric oxide at this point in the process avoids formation of a gelatinous, difficulty filterable iron precipitate which takes place above pH 7 and that would clog a filter used to remove the precipitates.

Using a 1,000,000 pound per month lead utilization lead acid battery plant as a exemplary standard, the present method treats 1,200 gallons a day of waste water from the battery wash operation to be processed daily with 200 gallons being discharged to the sewer and 1,000 gallons being recycled for reuse.

Waste water collected from the lead acid battery plant wash lines is stored in sump 10 which has an outlet line 12 leading to common supply line 14. Pump 16 is interposed in line 12 adjacent sump 10. A calcium carbonate slurry tank 18 is joined to supply line 14 through the provision of a line 20 having a pump 22 therein.

As is best seen in FIG. 6, supply line 14 includes an outer pipe 24 which extends through the cylindrical wall section 26 of generally conical first treatment tank 28. Line 12 is coupled directly to pipe 24 while pipe 20 extends into the interior of pipe 24 and is coaxial with the latter in spaced relationship from the interior wall of the latter to define a cylindrical passage around the periphery of pipe 20. Line 20 terminates within the interior of treatment tank 28 at the innermost extremity of pipe 24.

A recycle line 32 connected to first treatment tank 28 at the zone of merger of cylindrical section 26 with the conical segment 34 of such tank has a pump 36 therein and leads to pipe 24 for communication with the latter between pipe 12 and the innermost end of pipe 24. A pH controller 39 in line 32 is connected with pump 22 to control delivery of calcium carbonate from supply tank 18 to first treatment tank 28. Line 38 extending downwardly into the spherical top section 40 of treatment tank 28 serves to introduce an oxidizing medium such as air into the body of liquid contained in tank 28. The lowermost end of air line 38 terminates in a tee having a series of openings in the horizontal portion thereof for delivery of air in bubble form into the interior of the tank 28. An upright baffle 42 (see FIGS. 1-3) within tank 28 and spanning cylindrical section 26 in bisecting relationship thereto is joined to the underside of spherical top section 40 and projects downwardly into the tank a distance somewhat below the lower circular line defined by the bottom of cylindrical section 26. Air line 38 extends into tank 28 through an open manhole on the same side of baffle 42 as supply line 14 (see FIGS. 2 and 3).

Line 44 joined to cylindrical section 26 of first treatment tank 28 and communicating with the interior thereof, leads to a holding tank 46. A feed line 48 joins the lower end of holding tank 46 with an open top final treatment tank broadly designated 50. A delivery pump 49 is interposed in feed line 48. The cylindrical sidewall 52 of tank 50 terminates at the lower peripheral edge thereof in a shallow coned bottom 54. Mechanical agitator 55 is provided in tank 50 for stirring the contents thereof. Line 56 communicating with the final treatment tank 50 at the bottom of conical section 54 and provided with a pump 58 therein leads to a solids removal line 60 connected to the lower conical section 34 of treatment tank 28. Line 60 has a pump 62 therein and terminates in direct overlying relationship to a solids filter broadly designated 64.

As depicted schematically in FIG. 7, the solids filter 64 includes an underlying, angle bottom liquid collector 66 which receives an endless belt conveyor 68 which supports a disposable filter paper 70. Thus, solids deposited onto the filter paper supported by conveyor 68 are directed into the container 72 while liquid draining through the filter paper is returned to outlet line 12 via line 74 having a pump 76 therein.

Feed tank 78 for a slurry of calcium carbonate and calcium hydroxide has a delivery line 80 joined to the lower end thereof which feeds into the open upper end of final treatment tank 50. Pump 82 is interposed in line 80. Recycle line 86 extending from the tank 50 at the

lower periphery of the cylindrical section 52 thereof loops back over the top of tank 50 and delivers recycle liquid into the open end thereof. A pump 88 is provided in line 86 while pH controller 90 is operably connected to pump 82 for controlling delivery of slurry from feed tank 78 into tank 50. Flocculant storage tank 92 has a delivery pipe 94 joined thereto which extends to the open top of tank 50 and is provided with a feed pump 96 therein.

An outlet line 98 having a pump 100 extends from the lower part of final treatment tank 50 to the inlet of an upright sand filter 102. An outlet line 104 extending from the lower end of filter 102 is joined to the inlet of a final filter 106 which has an outlet pipe 108 leading to the sewer. Valve 110 in line 104 permits selective direction of waste water into recycle storage tank 112 via line 114. Recycle tank 112 is in turn connected to the pressure wash system 116 via line 118. Filter purge line 120 joins the sand filter 102 to the top spherical section 40 of first treatment tank 28. Backwash water is supplied to the lower end of sand filter 102 by a fresh water supply line 122.

#### Operation of Preferred Embodiment

During the manufacture of lead acid batteries, it is conventional to wash the batteries prior to packaging. Generally speaking, the batteries are supported by a conveyor which transports the units through a spray wash where they are subjected to three separate high-pressure spray washes. The wash water removes residual lead and sulfuric acid whereupon the batteries are blown dry. Waste water collected from the washing operation is directed via floor drains to the sump 10 along with the water used to wash the plant floor for treatment to permit recycling thereof to the wash water systems, with a certain proportion allowed to flow into a sewer line. The waste water is intermittently pumped via line 12 into the first treatment tank 28 through supply line 14. The waste water from pump 10 is generally saturated with lead sulfate and often contains suspended  $PbSO_4$ . Total lead in the waste water can run as high as 500 ppm. In addition, the waste water collected in sump 10 typically contains about 1%  $H_2SO_4$  giving the water a pH below 2. Iron is normally present at levels about 50 ppm while copper can be as high as 10 ppm. For a plant consuming about 1,000,000 lbs. of lead per month, the exemplary lead acid battery wash and floor wash systems will generate about 1,200 to 1,300 gallons per day of waste water collected in sump 10. The temperature of such water will be approximately the ambient temperature of the plant.

For a typical wash water system as described, treatment tank 28 should have a working capacity of approximately 1,300 gallons. Treatment of waste water in the tank 28 can be accomplished on a continuous or batch basis. Waste water collected in sump 10 is pumped to vessel 28 via line 12 until the treatment tank is substantially filled with the waste water to be treated. Thereafter, delivery of water to the treatment tank may be on an intermittent basis or substantially continuously, if the flow is controlled such that there is adequate residence time of the waste water in tank 28 to assure optimum precipitation of calcium sulfate and heavy metal carbonates which settle out of the solution and are removed from the tank via line 60. In order to assure collection of solids at the bottom of the tank 28, inclination of the conical wall 34 at an angle of about 30° has been found to produce satisfactory results. The divider baffle 42 desirably is of a depth such that it is connected

to and extends from the spherical top 40 of the tank to a horizontal line about 4 inches below the circular, circumferentially extending lower margin of the cylindrical section 26 of tank 28, thus dividing the upper part of the treatment tank 28 into a treatment section 28a (i.e. the upper portion of the tank 28 into which the waste water is directed via line 14) and a settling section 28b (i.e. the upper portion of the tank 28 from which the liquid is allowed to egress from the tank through line 44; see FIG. 3).

Air is sparged continuously into the interior of treatment tank 28 through line 38 to effect some mixing of the liquid contained in the tank and to also provide an adequate supply of an oxidizing agent (in this case  $O_2$ ) to convert the iron contained in the waste water to ferric oxide ( $Fe_2O_3$ ). However, the major mixing of liquid in treatment tank 28 is provided by recirculation of liquid through line 32 by pump 36.

Calcium carbonate and recycled waste water are mixed in supply tank 18 and the slurry thus produced is pumped via line 20 into the interior of treatment tank 28 extending coaxially through pipe 24. Operation of pump 22 controlling delivery of calcium carbonate slurry from supply tank 18 to first treatment tank 28 is under the control of pH metering unit 39. Although the specific concentration of the calcium carbonate in supply tank 18 is not highly critical, a 12% by weight slurry of the calcium carbonate may readily be pumped into the interior of the vessel 28. A normal rate during operation of pump 22 has been found to be about 80 pounds of calcium carbonate per 1,000 gallons of waste water. The amount required may vary within relatively large limits and depends in large measure on how well sulfuric acid spills are controlled in the lead acid battery manufacturing facility. The main consideration is that the amount of calcium carbonate furnished to first treatment tank 28 and the period of delivery thereof to the treatment vessel be controlled such that the pH of the waste water contained in treatment tank 28 be maintained at a pH of from about 4.8 to 5. Thus, controller 39 should be set up such that it initiates operation of pump 22 to deliver calcium carbonate slurry to the interior of first treatment tank 28 when the pH of the recycle water flowing through pipe 32 reaches a level of about 4.8, and then functions to discontinue energization of pump 22 when the pH of the solution in the recycle line 32 rises to a level of about 5.0. The calcium carbonate reacts with sulfuric acid in the waste water to produce calcium sulfate which precipitates from the solution as  $CaSO_4 \cdot 2H_2O$ . In addition, heavy metals present in the waste water such as lead, copper and zinc react with the calcium carbonate to form carbonate precipitates which, along with the calcium sulfate settle to the bottom of the tank 28 for removal via line 60. The majority of the carbonate however reacts with the sulfuric acid and is expelled as  $CO_2$  gas.

From one to three times per day solids which have collected in the lower conical section 34 of tank 28 are removed via line 60 during operation of pump 62. The high solids content liquid delivered from line 60 is deposited on the filter paper 70 of filter 64 where such solids are directed by conveyor 68 into the container 72. For most efficient operation of the filter 64, the unit is preferably of the type causing a vacuum to be imposed on the conveyor belt 68 with removed water being directed via line 74 back to supply line 12 through the action of pump 76. Solids collected in container 72

(which for example may be a 55 gallon drum) are shipped to a landfill.

By virtue of the fact that the waste water in first treatment tank 28 is not only recirculated but also must flow beneath the lower edge of baffle 42 from reaction zone 28a to the quiescent zone 28b, there is efficient interreaction between the calcium carbonate and the sulfuric acid and heavy metals contained in the waste water. Liquid overflow from treatment tank 28 leaves the latter via line 44 and is directed into the hold tank 46 by gravity flow. The pH of the waste water leaving vessel 28 is in the order of 5 to 7 because the calcium carbonate continues to react in the static section 28b of treatment tank 28. The soluble lead, copper and zinc in the waste water leaving first treatment tank 28 have been found to be less than 0.2 ppm but the totals may be above 10 ppm because of fine particles that are entrained in the liquid. On the other hand, the soluble iron content has been determined to be less than about 0.5 ppm if oxidation has been properly carried out in the reaction zone 28a of tank 28. Desirably, hold tank 46 is of a size to hold about 1,500 gallons of solution where tank 28 has a capacity of 1,300 gallons (although an emergency overflow hold tank of an additional 1,500 gallons capacity may be provided in association with hold tank 46 if additional hold capacity is desired).

Preferably, the contents of the hold tank 46 are pumped once a day into the final treatment tank 50 via line 48. An approximate 12% by weight solids slurry of a mixture of 20%  $\text{CaCO}_3$  and 80%  $\text{Ca(OH)}_2$  (providing a mole ratio of carbonate to hydroxide of about 1:10) is mixed in feed tank 78 and pumped into final treatment tank 50 through line 80 under the control of pump 82. Again the 12% concentration of the calcium carbonate-calcium hydroxide mixture may readily be pumped through line 80 to the tank 50. The ratio of  $\text{CaCO}_3$  to  $\text{Ca(OH)}_2$  is such as to provide an excess of carbonate ions in final treatment tank 50. Calcium hydroxide is necessary to raise the pH to the desired level in the final treatment. Under normal operating conditions there is little, if any, reaction of calcium carbonate in the treatment tank 50. The solubility of lead hydroxide (2.1 ppm as lead) is too high to meet specifications; thus, steps are taken to preclude formation of lead hydroxide in the process. Typical usage of  $\text{Ca(OH)}_2$  is no more than about 8 pounds per 1,000 gallons of waste water.

Mixing of the solution in treatment tank 50 is assured by a slow speed agitator in the nature of a propeller mixer 55. In addition, the solution in treatment tank 50 is recirculated through line 86 under control of pump 88. The pH controller 90 functionally coupled to pump 82 controls the operation of the latter to feed the  $\text{CaCO}_3$ - $\text{Ca(OH)}_2$  slurry into treatment tank 50 upon demand. The controller is adjusted between a pH of 8.0 and 9.0. When the pH of the solution reaches 9.0, operation of pump 82 is discontinued, whereupon the contents of treatment tank 50 are mixed for at least about 1 hour and then an anionic polyelectrolyte flocculant contained in tank 92 is pumped via line 94 into the final treatment tank 50. A preferred flocculant giving desirable results has been found to be Mitfloc 51 supplied by Mitco Incorporated, Grand Rapids, Mich. After approximately 1 minute of mixing, the system is shut off and the contents of final treatment tank 50 are allowed to settle for from 2 to 24 hours and most preferably about 20 hours.

Heavy metal carbonates and any residual calcium sulfate collected in the bottom of final treatment tank 50

are returned to line 60 via line 56 under the control of pump 58 for filtering of such solids in association with those received from first treatment tank 28. The liquid is thereby returned to first treatment tank 28 through line 74, line 12 and supply line 14.

The clear supernatant resulting from quiescent settling of solids from the waste water in tank 50 is pumped via transfer line 98 under the control of pump 100 to sand filter 102 to remove suspended particles in the waste water. Backwashing of the sand filter 102 should be carried out about once a week with water being supplied for this purpose through line 22 and the resulting solids removed being directed back into first treatment tank 28 through line 120.

The discharge from sand filter 102 is directed via line 104 to a three way valve 110 with one line 104 therefrom going to the final filter 106 while another line 114 returns water to the recycle tank 112 which is connected to the pressure wash system 116 through line 118. The waste water treated each day is divided such that about an average of 80% thereof is returned to the pressure wash system 116 while the remaining 20% goes to the final filter 106 for ultimate discharge to the sewer via line 108. The treated waste water leaving final filter 106 is normally well within regulations established by the EPA. However, final filter 106 desirably containing a 5 micron bag filter is a safety measure if a malfunction of sand filter 102 occurs for some reason, or if some particularly fine material is not removed by settling in the final treatment tank and passed through filter 102. In a preferred arrangement, recycle tank 112 is of dimensions holding approximately 4 days of water for supply to the plant pressure wash system 116.

Oxidation of iron contained in the waste water while the latter is held in tank 28 prevents formation of a hard to filter iron precipitate in the final treatment tank 50. If unoxidized iron is allowed to reach tank 50, filtration problems can occur above pH 7 (noting in this respect that the pH of the waste water in tank 50 is desirably at least about 8 up to about 9),  $\text{Fe(OH)}_3$  which forms can be very difficult to filter. Air sparging of the waste water in the first treatment tank 28 not only permits utilization of the air as an agitation medium, but also assures conversion of the iron to  $\text{Fe}_2\text{O}_3$  which precipitates and may be readily filtered out in the filter 64.

A particularly important feature of the present process is the fact that a relatively inexpensive commodity of commerce (limestone, i.e. calcium carbonate) may be employed to effect removal of sulfuric acid and heavy metals from the battery acid plant waste water. Limestone not only may be obtained at a reasonable cost but is available in a particle size that assures efficient reaction thereof with the acid and heavy metals in the waste water. Typically, calcium carbonate sold as paint pigment or an animal feed additive has a particle size of approximately 5 microns. Desirably, the calcium carbonate is of a particle size such that at least about 90% will pass through a 100 mesh screen.

In spite of the low solubility of  $\text{CaCO}_3$  in water, the present process takes advantage of the fact that calcium carbonate becomes more soluble in a solution saturated with  $\text{CO}_2$ . Since the reaction of  $\text{H}_2\text{SO}_4$  with  $\text{CaCO}_3$  results in release of  $\text{CO}_2$  thus saturating with the solution of carbon dioxide, the carbonate ion is present in sufficient supply to assure precipitation of lead and other heavy metals as carbonates. It was therefore unexpectedly found, contrary to the teachings of the prior art as for example, indicated in the above-referenced

EPA Development Document, that effective removal of lead and other heavy metals may be accomplished at a pH in the order of 5.

Although best results are obtained at the lead cost using limestone ( $\text{CaCO}_3$ ) as the precipitating agent for the sulfuric acid and heavy metals in the waste water, other alkali metal and alkaline earth carbonates may be employed. Suitable reactants in this respect would include lithium carbonate, sodium carbonate, potassium carbonate, magnesium carbonate, and barium carbonate.

In the second treatment tank 50, wherein calcium hydroxide in association with calcium carbonate is added to the waste water,  $\text{Ca}(\text{OH})_2$  is the additive of choice for cost reasons but again other alkali metal and alkaline earth hydroxides would be usable such as lithium hydroxide, sodium hydroxide, potassium hydroxide, and barium hydroxide. Use of salts that form soluble metal sulfates will cause a buildup of that metal salt in the water thus precluding recycling of the treated waste water even though lead, copper and zinc are removed to desirable sewer discharge levels.

Results obtained from practicing the process of this invention on an actual plant basis may be summarized as follows (21 days actual plant operation):

Day	Lot No.	Gals.	Copper		Lead		30
			To Sewer	pH	PPM	To Sewer	
1	1003		8.37	0.10		0.12	
2	101R		7.89	0.16		<0.10	
3	102R		8.07	0.13		0.14	
4	103S	130	8.12	0.17	0.00018	0.10	0.00011
5	105R		9.16	0.21		0.37	
6	106S	910	10.62	0.07	0.00053	<0.10	0.00076
7	107R		9.02	0.05		<0.10	
8	108R		7.25	0.12		<0.10	
9	109S	270	9.38	0.10	0.00023	<0.10	0.00023
10	110S	210	7.30	0.16	0.00028	<0.10	0.00018
11	111R		9.44	0.14		<0.10	
12	112S	730	6.51	0.12	0.00073	0.19	0.00116
13	113R		6.93	0.06		0.14	
14	114R		6.98	0.05		<0.10	
15	115R		7.11	0.08		0.19	
16	116R		7.05	0.02		<0.10	
17	117S	240	8.42	0.02	0.00004	<0.10	0.00020
18	118S	310	7.13	0.03	0.00008	<0.10	0.00026
19	119S	30	7.31	0.03	0.00001	<0.10	0.00003
20	120S	80	8.01	0.03	0.00002	<0.10	0.00007
<b>TOTAL</b>		2910			0.00210		0.00298

This Period:

Lead Consumed: 725,384 lbs.

Days Production: 21

EPA Allowable Discharge:

-continued

5	Copper		Lead	
	0.04974 lbs/day	0.55129 lbs/period	0.01105 lbs/day	0.10881 lbs/period
EPA Discharge Limits (Lbs. Per Million Lbs. of Lead Consumed):				
10	Copper		Lead	
Daily		1.44	0.32	
Monthly		0.76	0.15	

I claim:

1. In a method of treating industrial waste water from a lead acid battery manufacturing plant contaminated with environmentally unacceptable amounts of sulfuric acid along with lead and copper to lower the concentration thereof to levels permitting discharge of the water to a sewer, the improved steps of:

adding an amount of an alkaline earth carbonate selected from the group consisting of calcium, magnesium and barium carbonate and mixtures thereof to the waste water to initially raise the pH thereof to a level which is especially conducive to precipitation of lead and copper carbonate and formation of an alkaline earth sulfate from the sulfuric acid, the amount of alkaline earth carbonate added to the waste water being sufficient to raise the pH of the waste water to a level of about 4 to about 5 to effect formation of an adequate quantity of alkaline earth sulfate and precipitation of lead and copper carbonate such that upon removal of the precipitates, waste water may be discharged to the sewer which meets environmental restrictions imposed on the discharge;

35 a mixture of alkaline earth carbonate and alkaline earth hydroxide is added during the second addition and removing lead and copper carbonate precipitates from the waste water prior to discharging the latter to the sewer.

40 2. In a method of treating industrial waste water as set forth in claim 1, wherein adjusting the pH of the waste water following the addition of the alkaline earth carbonate thereto, to a level above about 7 by addition of an alkaline earth hydroxide before effecting removal of precipitates from the waste water thereby preventing the redissolution of suspended lead and copper carbonates; and the mole ratio of carbonate to hydroxide in the second addition to the waste water is about 1 to 10.

45 3. In a method of treating industrial waste water as set forth in claim 1, wherein the step of adjusting the pH of the waste water to a value above about 7 with an alkaline earth hydroxide is carried out in a manner to cause the waste water to have a pH of about 8 to 9 prior to discharge of the waste water to the sewer.

55 \* \* \* \*



# UNITED STATES COURT OF FEDERAL CLAIMS

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CHEMICAL SEPARATION TECHNOLOGY, )  
INC. and SANFORD M. STEVENSON, )  
  )  
  )  
Plaintiff, )  
  )  
  )  
v. ) No.: 97-21C  
  )  
  )  
THE UNITED STATES, )  
  )  
  )  
Defendant. )

## BENCH DECISION VOLUME II, MARKMAN HEARING

Pages: 297 through 427  
Place: Pittsburgh, Pennsylvania  
Date: November 14, 2000

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1 your briefs, the appendices, my review of the case law, and  
2 my consideration, obviously, of the intrinsic, and to the  
3 extent relevant, extrinsic evidence to make rulings with  
4 respect to the construction of the claims in the 800 and in  
5 the 497 patent.

6           My intent is to go through these rulings basically  
7 one at a time. My expectation is not to have each of you  
8 comment on whether you agree or disagree with my ruling. I  
9 think the ruling at this point is going to stand. There is  
10 one exception to that, and I will point that out when we get  
11 to that point. And so what I think what I am going to do is  
12 to go right ahead into this dealing with the elements in the  
13 claims one by one.

14           Any question about how we are going to proceed,  
15 Mr. Tarasi?

16           MR. TARASI: No, Your Honor.

17           THE COURT: You, Mr. Elliot?

18           MR. ELLIOT: No, Your Honor.

19           THE COURT: All right, then, let's begin with the  
20 800 patent, Claim 1, the phrase "removing." My ruling is  
21 that that phrase means "to take away or eliminate."

22           The next portion of Claim 1 at issue is metal  
23 compounds. By agreement of the parties, that portion of the  
24 claim is construed to mean "precipitated or suspended  
25 compounds of metal."

1           The next portion of Claim 1 of the 800 patent  
2 indicates "selected from iron, manganese, aluminum, zinc,  
3 copper, lead, arsenic and chromium." By agreement of the  
4 parties that language shall be construed to mean "selected  
5 from iron, manganese, aluminum, zinc, copper, lead, arsenic  
6 and chromium, either alone or in combination with themselves  
7 or other materials and in any initial form so long as they  
8 are precipitated or suspended."

9           The next portion of the 800 patent being construed  
10 is the part in Claim 1 dealing with the word "comprising".  
11 Construction placed on that term is "comprising at least" by  
12 agreement of the parties.

13           The next portion of Claim 1 of 800 to be construed  
14 is "adjusting the pH of the water to from about to about  
15 12." My ruling is that that portion of the claim is not  
16 indefinite, and that it refers to "Bringing to a pH point  
17 within the range specified that optimizes the precipitation  
18 of metals, said point to be determined by reasonable  
19 experimentation."

20           So again, on some of these in which we don't have  
21 a reference in the brief let me just repeat it to make sure.  
22 I determined that this portion of Claim 1 is not definite  
23 and that it refers to "Bringing to a pH point within the  
24 range that optimizes the precipitation of metals, said point  
25 to be determined by reasonable experimentation."

1                   The next portion of Claim 1 at issue involves  
2    "aerating the waste water." My construction of that is it  
3    means "supply air and other gaseous oxidants to the waste  
4    water."

5                   The next portion of Claim 1 at issue, "agitating  
6    the waste water." By agreement of the parties this element  
7    is deemed to means "includes agitation by any means,  
8    including by aeration and by a mixer."

9                   The next portion of Claim 1 to be construed  
10    involves that portion of Claim 1 stating, "Aerated and said  
11    reaction tank to provide a dissolved oxygen concentration at  
12    from about 0.01 pounds per hour to about 70 pounds per  
13    hour." I also want to construe in conjunction with that  
14    portion of Claim 1 that deals with "about 50 gallons per  
15    minute to about 500 gallons per minute and about 50  
16    milligrams per liter to about 1,000 milligrams per liter."

17                  Based upon the consideration of the language,  
18    based upon the expert testimony in particular, and so this  
19    is one in which I am relying on extrinsic evidence, I  
20    determined that this refers to a rate of aeration, and that  
21    the limitations -- excuse me -- that the ranges expressed in  
22    terms of "50 gallons per minute to about 500 gallons per  
23    minute and about 50 milligrams per liter to about 1,000  
24    milligrams per liter" are not intended as limitations on the  
25    patent.

1                   The next portion of Claim 1 to be construed  
2    involves "flocculating agency polymer." By agreement of the  
3    parties that is construed to include a polyelectrolyte.

4                   The next portion of Claim 1 of the 800 patent to  
5    be construed involves "a group consisting of cationic and  
6    anionic polymers." By agreement of the parties that phrase  
7    is construed as "limited to cationic and anionic polymers,  
8    but includes any cationic or anionic polymer."

9                   Moving now to Claim 2 of the 800 patent, the  
10   phrase "further dewatering the floccules separated in step  
11   E." By agreement of the parties that phrase means  
12   "processing the floccules separated in step E in a second or  
13   later stage."

14                   The next portion of the 800 patent to be construed  
15    is Claim 9, the phrase "polishing means."

16                   Could I get you to pause for a second, please?

17                   (Pause.)

18                   THE COURT: All right, going to Claim 9, the  
19    phrase "polishing means." By agreement of the parties that  
20    is construed to mean "limited to a polishing pond, a  
21    settling pond and a polishing tank and their equivalents."

22                   Claim 21, still in the 800 patent, "use for  
23    primary clarification purposes" is the phrase to be  
24    construed. By agreement of the parties that means "to  
25    include in a first-stage clarifier." Excuse me. That means

1 to be construed as "in a first-stage clarifier."

2 Still in the 800 patent Claim 22, "used for  
3 settling purposes." By agreement of the parties this means  
4 "in a settling pond or tank."

5 Claim 23 of the 800 patent, the Court is going to  
6 reserve ruling on Claim 23. I believe that there are  
7 serious concerns regarding the definiteness or in fact  
8 indefiniteness of this claim, and in particular, I feel the  
9 need to review more closely the experts' oral testimony, so  
10 I'm going to reserve ruling on this claim, and we will  
11 discuss at the conclusion of the ruling how we will deal  
12 with that with respect to the impending trial.

13 Moving now to the 497 patent, the portion of Claim  
14 one that states "aerator means having a shaft extending  
15 therefrom into said reaction vessel, said shaft having a  
16 discharge end for discharging oxidant." By agreement of the  
17 parties this is a means plus function. It refers to  
18 discharging air and other gaseous oxidants.

19 The portion of the language in this claim that  
20 deals with "aerator means" is illustrated by item 6 in  
21 Figure 3. The remainder of this clause, this element  
22 dealing with "having a shaft extending therefrom into said  
23 reaction vessel, said shaft having a discharge end for  
24 discharging oxidant" is illustrated items 15, 16 and 17 in  
25 Figure 3.

LOUIS M. TARASI, JR.  
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July 20, 2001

**VIA FACSIMILE & REGULAR MAIL**

The Honorable Francis M. Allegra  
United States Court of Federal Claims  
717 Madison Place, N.W.  
Washington, DC 20005

**RE: Chemical Separation Technology, Inc., and Sanford Stevenson, v.  
The United States of America, No. 97-21 C**

Dear Judge Allegra:

As an officer of the Court, and in fairness to this Court and opposing counsel, I feel it is necessary to bring the following to the Court's attention. I write to advise you that Mr. Stevenson has recently received notice from the USPTO of the allowance of his Application Serial No. 09/652,272 for a United States Patent. I bring this to your Honor's attention because Application Serial No. 09/652,272 is a continuing application from the application on which U.S. Patent No. 5,370,800 was based and thereby entitled to the same priority date of May 25, 1993.

The allowed claims are essentially the same as those of the '800 patent except that they apply specifically to copper rather than to the Markush group of metals recited in claim 1 of the '800 patent.

The significance of the allowance of this Patent Application to the present litigation, and your Honor's consideration of the United States invalidity contentions, is that the prior art raised by Defendants, viz., the EPA manual, Inglis patent and the Summitville site bid, were all specifically disclosed to the U.S. Patent and Trademark Office (USPTO) in this recently granted application. The issues under 35 USC § 102 and § 103 in the claims presented to the USPTO, and essentially the same as those before your Honor in the present matter. The decision of the USPTO to allow the claims in this application over the very prior art cited by Defendants we believe is both relevant and persuasive that the 800 Patent is valid under §§ 102 & 103 given the presumption of validity that attaches to a U.S. Patent.

The Honorable Francis M. Allegra

July 20, 2001

Page 2

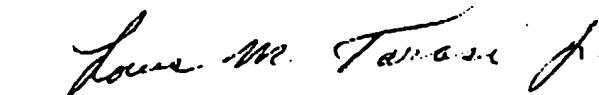
For instance, the broadest claim allowed in the application (claim 7, which will be renumbered as claim 1 when the patent issues) reads as follows:

7. A method for removing metal compounds comprising copper metal compounds from waste water comprising the steps of:
  - (a) adjusting the pH of the waste water to from about 5 to about 12;
  - (b) aerating the waste water;
  - (c) agitating the waste water, where steps (a), (b) and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a copper metal concentration of from about 50 mg./L to about 1,000 mg./L;
  - (d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said copper metal compounds to form floccules; and
  - (e) then separating said floccules including said copper metal compounds from the water.

We have requested, and will be receiving, the a certified copy of the prosecution history and patent that relates to Application Serial No. 09/652,272. We anticipate the certified copies to be available within 30 days. Upon receipt, we will forward a certified copy of the same to your attention, and request that you take judicial notice of these facts, disclosed by the prosecution history and patent, pursuant to F.R.E. 201.

Respectfully Submitted:

TARASI, TARASI & FISHMAN, P.C.



Louis M. Tarasi, Jr., Esquire

CC:

Cameron Elliot, Esquire

LOUIS M. TARASI, JR.  
CERTIFIED CIVIL TRIAL ADVOCATE BY THE NATIONAL  
BOARD OF TRIAL ADVOCACY. A PENNSYLVANIA  
SUPREME COURT APPROVED AGENCY.

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July 24, 2001

**VIA FEDERAL EXPRESS**

Cameron Elliot, Esquire  
Commercial Litigation Branch  
Civil Division  
Department of Justice  
Washington, DC 20530

**RE: CST & Stevenson v. U.S.  
97-21 C**

Dear Mr. Elliot:

In response to your correspondence of July 20<sup>th</sup>, enclosed, please find Mr. Stevenson's file relative to the prosecution of his Application Serial No. 09/652,272, excluding the prior art.

I have copied the Court on this correspondence, and included the enclosures. Upon receipt of the certified copies of the prosecution history and patent, I will forward a copy to you at the same time I provide the original to the Court.

Sincerely,

TARASI, TARASI & FISHMAN, P.C.

  
C. William Kenny, Esquire

Enclosure

cc:

The Honorable Francis M. Allegra (w/enclosure)

Rec'd 7/25/01 MM



# THE UNITED STATES OF AMERICA

**TO ALL TO WHOM THESE PRESENTS SHALL COME:**

**UNITED STATES DEPARTMENT OF COMMERCE**

**United States Patent and Trademark Office**

March 14, 1997

**THIS IS TO CERTIFY THAT ANNEKED IS A TRUE COPY FROM THE RECORDS  
OF THIS OFFICE OF THE FILE WRAPPER AND CONTENTS OF:**

**APPLICATION NUMBER: 08/072,418**

**FILING DATE: May 25, 1993**

**PATENT NUMBER: 5,370,800**

**ISSUE DATE: December 06, 1994**

**TITLE OF INVENTION:**

**METHOD FOR REMOVING METAL COMPOUNDS FROM WASTE WATER**

**INVENTOR(S):**

**STEVENSON, SANFORD M.**



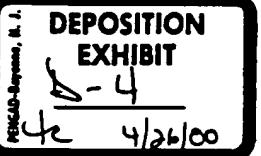
**By Authority of the  
COMMISSIONER OF PATENTS AND TRADEMARKS**

*T. Lawrence*

**T. LAWRENCE  
Certifying Officer**

DO24718

**EXHIBIT**



UTILITY SERIAL NUMBER	372418	PATENT DATE	DEC 07 1993	PATENT NUMBER
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5370800

SERIAL NUMBER 08/072,418	FILING DATE 05/25/93	CLASS 210	SUBCLASS 710	GROUP ART UNIT 1206	CHAMBER 1
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SANFORD M. STEVENSON, McMURRAY, PA.

\*\*CONTINUING DATA\*\*\*\*\*  
VERIFIED

f44

III

\*\*FOREIGN/PCT APPLICATIONS\*\*\*\*\*  
VERIFIED

f44

FOREIGN FILING LICENSE GRANTED 07/19/93

\*\*\*\*\* SMALL ENTITY \*\*\*\*\*

Foreign priority claimed 35 USC 119 conditions met	<input type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	AS FILED	STATE OR COUNTRY PA	SHETS DRAWN 2	TOTAL CLAIMS 38	INDEP. CLAIMS 3	FILING FEE RECEIVED \$553.00	ATTORNEY'S DOCKET NO. 600141
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Verifier and Acknowledger (Signature and Address)

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DANIEL J. LUNG  
2230 KOPPERS BUILDING  
436 SEVENTH AVENUE  
PITTSBURGH, PA 15219

METHOD AND APPARATUS FOR REMOVING METAL COMPOUNDS FROM WASTE WATER

U.S. DEPT. OF COMMERCE &amp; THE GPO—PTO-488A, 500-1400

6-23-94

PARTS OF APPLICATION FILED SEPARATELY		L. Johnson Applicant Examiner	
NOTICE OF ALLOWANCE MAILED	6/23/94	CLAIMS ALLOWED	
ISSUE FEE	Assistant Examiner	Total Claims 25	Pat. Claims 1
Amount Due \$2500.00	Date Paid 7-25-94	Drawings	
		Sheets Drawn 2	Page Drawn 4
		Primary Examiner J. L. Johnson	Print Pg. 1
Label Area		ISSUE BATCH NUMBER B 91	
PREPARED FOR ISSUE			
WARNING: The information contained herein may be restricted. Unauthorized disclosure may be prohibited by law, including Title 35, Sections 122, 181 and 365. Possession outside the U.S. and its possessions is restricted to authorized employees and contractors only.			

PTO-488A  
Rev. 6-92

D024719

00 072418

MAY 25, 1993

THE COMMISSIONER OF PATENTS AND TRADEMARKS  
Washington, D.C. 20231

Sir:

Transmitted herewith for filing is the patent application of

**Inventor** SANFORD M. STEVENSON

**For:** METHOD AND APPARATUS FOR REMOVING METAL COMPOUNDS  
FROM WASTE WATER

**Enclosed are:**

2 sheets of INFORMAL drawings.

An assignment of the invention to \_\_\_\_\_

A certified copy of a \_\_\_\_\_ application.

Declaration and Power of Attorney for a Patent Application

A verified statement to establish small entity status under  
37 CFR 1.9 and 37 CFR 1.27.

PETITION TO MAKE SPECIAL UNDER 37 CFR §1.102(c)

The filing fee has been calculated as shown below

(Col. 1) (Col. 2)

FOR:	NO. FILED	NO. FILED
<b>BASIC FEE</b>		
<b>TOTAL CLAIMS</b>	38 -20=	* 18
<b>INDEP CLAIMS</b>	- 3=	* -0-
<b>MULTIPLS/DEPENDENT CLAIM PRESENTED</b>		

SMALL ENTITY		OTHER THAN A SMALL ENTITY	
		<u>OR</u>	
	\$355	<u>OR</u>	\$710
X 11	\$198.00	<u>OR</u>	
X 37	\$ -0-	<u>OR</u>	X 22
X 115	\$	<u>OR</u>	X 74
		<u>OR</u>	X 230
	<b>TOTAL</b>	<b>TOTAL</b>	\$
	\$553.00		

A check in the amount of \$553.00 to cover the filing fee is  
enclosed.

A check in the amount of \$ \_\_\_\_\_ for the Government Fee for recording  
an Assignment.

Respectfully submitted,



DANIEL J. STEVENSON

ATTORNEY FOR APPLICANT

REGISTRATION NO. 29404

(412) 471-9633 FAX NO. (412) 765-1583

0024720

EXPRESS MAIL TB208045849US

08 072418

PATENT APPLICATION SERIAL NO. 08 072418

U.S. DEPARTMENT OF COMMERCE  
PATENT AND TRADEMARK OFFICE  
FEES RECORD SHEET

050 AT 06/24/93 08072418

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## METHOD AND APPARATUS FOR REMOVING METAL COMPOUNDS FROM WASTE WATER

### Background of the Invention

The present invention relates to the treatment of waste water, and, in particular, to the treatment of water to remove various precipitated or suspended metal compounds therefrom.

Runoffs from a number of industrial operations such as electrical power plants, steel plants and mines are known to be contaminated with various metal compounds including iron, manganese, aluminum, zinc, copper, lead, arsenic and chromium. Such contaminants may pose a serious environmental problem.

Methods heretofore used to remove such contaminants have included the use of additional lime, soda ash or other neutralizing agents and the use of holding ponds or clarifying tanks. Such methods have not, however, been entirely satisfactory because of the lengthy periods of time which they would generally be required to effect treatment.

### Summary of the Invention

It is therefore an object of the present invention to provide an improved method and apparatus for removing precipitate or suspended metal compounds from waste water.

In this method the pH of the water is first adjusted from 6 to 10. Preferred neutralizing agents are sodium hydroxide, and anhydrous ammonia when the waste water is overly acidic or sulfuric acid or hydrochloride acid when it is overly basic. The water is also aerated to a dissolved oxygen

concentration of from .01 to 70 lb./hr. Neutralization and aeration may preferably be done simultaneously. A polymeric flocculating agent is then added to the water. The metal compounds are then flocculated, and the flocculated metal compounds are separated from the water by means of a rotary drum thickener, clarifier or other suitable means. A preferred flocculating agent is an anionic or cationic polymer wherein the use of an anionic polymer would be preferred for primary clarification or setting purposes while the cationic polymer would be preferred for dewatering purposes. The flocculated metal compounds are then further dewatered in a belt filter press or other suitable apparatus.

Brief Description of the Drawings

The present invention is further described with reference to the accompanying drawing in which:

Figure 1 is a schematic drawing of apparatus used to carry out one preferred embodiment of the method of the present invention;

Figure 2 is a schematic drawing of apparatus used to carry out another preferred embodiment of the method of the present invention;

Figure 3 is a schematic drawing of apparatus used to carry out a third preferred embodiment of the method of the present invention; and

Figure 4 is a schematic drawing of apparatus used to carry out a fourth preferred embodiment of the method of the present invention.

Detailed Description

Referring to Figure 1, the raw water source is shown at numeral 10. Waste water is removed from this source in line 12. Neutralizing agent tank 14 is connected by line 16 to reaction tank 18 where water from line 12 is simultaneously mixed with neutralizing agent, aerated and agitated. A preferred embodiment of the reaction tank 18 is described in U.S. Patent No. 4,749,497 although it has now been found that in some cases the apparatus disclosed in that patent may be advantageously modified by the removal of its baffles and the ~~velocation~~ <sup>location</sup> of the aeration mixing unit to a 45° angle to the vertical plane of the reaction tank.

<sup>01</sup>  
B The rate of aeration would normally be from about ~~10~~ lbs./hr. to about 70 lbs./hr. for a metals concentration of 50 mg./l. to 1,000 mg./l. at a raw water input flow rate of 50 gal./min. to 500 gal./min. The pH of the water is adjusted to between 5 and 12 and preferably to between 6 and 9. It will be understood that for regulatory reasons if the pH is adjusted to above 9, it will ordinary be necessary to reduce the ~~pH~~ to below that level on completion of the process described herein. The water is then removed in line 20 to rotary drum thickener 22. A flocculating agent from polymer tank 24 is also moved in line 26 to line 20 to be mixed with the water in rotary drum thickener 22 where the metal compounds are flocculated and separated. Such polymers

which are used for primary classification purposes are anionic polymers. NALCO 7767 is a suitable commercially available anionic polymer. Cationic polymers may be used for dewatering purposes and may be selected from the following commercially available polymers: PERCOL AC 737 and UNIFLOC 630. Water from the rotary drum thickener is removed in line 28 to polishing pond 30 from where outfall is removed in line 32. Water and flocculated metal compounds are removed from the rotary drum thickener in line 34. Additional polymer from polymer tank 36 is moved in line 38 to be mixed with water and flocculated metal compound in line 38 which are then moved to belt filter press 40 where the ~~flocculants~~ are dewatered and from which water is removed to the polishing pond in line 42.

Referring to Figure 2, waste water from a raw water source 110 is removed in line 112 to be mixed with neutralizing agent from tank 114 moved in line 116 to reaction tank 118. The neutralized, aerated and agitated water is then removed in line 120 to clarifier 122 to which flocculating agent from polymer tank 124 through line <sup>126</sup><sub>125</sub> is mixed. Water is removed from the clarifier in line 128 to polishing pond 130 from where outfall is released from line 132. Flocculated metal compound along with water is removed from the clarifier in line 134 from where is mixed with additional polymer from polymer tank 136 and line 138 and then dewatered in belt filter press 40 where additional water is removed and transported to polishing pond in line 142.

Referring to Figure 3, waste water from raw water source 210 is removed in line 212 and neutralizing agent from tank 214 is removed in line 216. The water is introduced to reaction tank 218 from where it is removed in line 220 to clarifier 222 after being mixed with flocculating agent from polymer tank 224 moved through line 226. Water from the clarifier is removed in line 228 to polishing pond 230 from where outfall is removed in line 232. Floccules containing metal compounds are removed with water from the clarifier in line 234. Additional polymer from polymer tank 236 is removed in line 238 to be mixed with the floccules which are then introduced to rotary drum thickener 239 and are then introduced to belt filter press 240 after mixed with additional polymer from tank 241 which is removed in line 243 and mixed with water from the rotary drum thickener removed through line 245. Water from the belt filter press is removed in line 242 and transformed with water from line 244 from rotary drum thickener 239 to the polishing pond.

Referring to Figure 4, waste water from raw water source 310 is removed in line 312 and mixed with neutralizing agent from tank 314 and line 316 in reaction tank 318 from where water is removed in line 320 to settling pond 322. Flocculating agent is introduced from polymer tank 324 through line 326. Water is removed from the settling pond in line 328 to polishing pond 330 from where outfall is removed in line 332. Floccules containing metal compound along with water are removed in line 334 and are mixed with additional polymer from tank 336.

introduced through line 338 and then dewatered in belt filter press 340 from which water is removed to the polishing tank in line 342. Alternatively, portions of the floccules, water and polymer mixture in line 334 are also diverted to borehole 344 and drying bed 346.

The method and apparatus of the present invention is further described with reference to the following example.

Example

350 GPM of waste water having a pH of 2, having dissolved metal concentrations of 10,000 ppm was first pumped from a collection area to a reaction vessel where it was aerated and agitated at a dissolved oxygen concentration of 3 lb./HP hour. Sodium hydroxide was added automatically as the neutralizing agent, at juxtaposition to the waste water, aeration and agitation point, to an adjusted pH of 8.5. The neutralized waste water is transferred to a flocculator reactor where a

← UNIFLOC 630 cationic polymer is added at a rate of 2 GPM. The instantaneous agglomeration of the neutralized waste water was transferred to a rotary drum thickener where water was filtered from the metal hydroxide sludge, where supernate water was removed to a polishing pond at a rate of approximately 300 GPM. Metal hydroxide sludge was removed from the rotary drum thickener at a rate of approximately 50 GPM to a belt filter press where a cationic polymer was added at a rate of 1 GPM. After processing through the belt filter press cake solids were

recovered at a 15% to 40% dry solids. Water leaving the polishing pond consistently had a metal ion concentration below EPA permitted limits.

Although the invention has been described with a certain degree of particularity, it is to be understood that the present disclosure has been made only as an example and that the scope of the invention is defined by what is claimed hereafter.

WHAT IS CLAIMED IS:

1. A method for removing metal compounds from waste water comprising the steps of:

- (a) adjusting the pH of the waste water to from about 5 to about 12;
- (b) aerating the waste water;
- (c) then adding a flocculating agent to the water and allowing floccules including metal compounds to form; and
- (d) then separating said floccules including metal compounds from the water.

2. The method of claim 1 wherein before step (c) there is added step (e) of agitating the waste water.

3. The method of claim 1 wherein there is added the further step (f) of further dewatering the floccules separated in step (d).

4. The method of claim 1 wherein in step (a) the pH is adjusted to from about 6 to about 9.

5. The method of claim 1 wherein steps (a) and (b) are carried out simultaneously.

6. The method of claim 1 wherein in step (a) the pH is adjusted by adding a neutralizing agent selected from sodium hydroxide, anhydrous ammonia, sulfuric acid and hydrochloric acid.

7. The method : wherein in step (b) the water is aerated to a dissolved concentration of about .01 lb./hr. to about 70 lb./hr.

8. The method of : wherein in step (c) the flocculating agent is a polymer.

9. The method of claim 8 wherein the polymer is selected from anionic or cationic polymer.

10. The method of claim 9 wherein the polymer is a cationic polymer which is used for dewatering purposes.

11. The method of claim 9 wherein the polymer is an anionic polymer which is used for primary clarification purposes.

12. The method of claim 9 wherein the polymer is an anionic polymer which is used for settling purposes.

13. The method of claim 8 wherein the polymer is added in a dilute concentration of from about 0.5% to about 1.5% by weight.

14. The method of claim 1 wherein after step (d) a portion of the separated water is removed to a polishing pond.

15. The method of claim 1 wherein in step (d) separation is conducted by means of a rotary drum thickener.

16. The method of claim 3 wherein additional flocculating agent is added to at least a portion of the waste water containing the flocculated metal compound separated in step (d).

17. The method of claim 16 wherein after the addition of the additional flocculating agent, the flocculated metal compound is dewatered in step (f) in a belt filter press.

18. The method of claim 16 wherein water removed in step (e) is removed to a polishing pond.

19. The method of claim 3 wherein in step (c) separation is conducted by means of a clarifier.

20. The method of claim 19 wherein additional flocculating agent is added to at least a portion of the flocculated metal compound separated in step (d).

21. The method of claim 20 wherein after the addition of the additional flocculating agent, the flocculated metal compound is dewatered in step (f) in a belt filter press.

22. The method of claim 21 wherein water removed in step (e) is removed to a polishing means.

23. The method of claim 1 wherein in step (d) separation is conducted by means of sequential treatment in a clarifier and a rotary drum thickener.

24. The method of claim 23 wherein additional flocculating agent is added after the clarifier and then again after the rotary drum thickener.

25. The method of claim 24 wherein after the additional flocculating agent, the flocculated metal compound is dewatered in step (f) in a belt filter press.

26. The method of claim 25 wherein the water removed in step (e) is removed to a polishing pond.

(5) 27. The method of claim 1 wherein in step (d) separation is conducted by means of a settling pond.

28. The method of claim 27 wherein additional flocculating agent is added after the settling pond.

(C) 29. The method of claim 28 wherein after the additional polymer is added the flocculated metal compound is dewatered in step (f) in a belt filter press.

30. The method of claim 24 wherein water removed in step (e) is removed to a settling pond.

31. The method of claim 1 wherein the separated metal compounds are selected from iron, manganese, aluminum, zinc, copper, lead arsenic and chromium.

32. An apparatus for removing metal compounds from waste water comprising:

- (a) means for adjusting the pH of the waste water;
- (b) means for aerating the waste water;
- (c) means for adding a flocculating agent to the water;
- (d) means for separating said floccules including metal compounds from the water.

33. The apparatus for removing metal compounds from waste water wherein the apparatus includes as an element (e) a means for agitating the waste water which is used in conjunction with elements (a) and (b).

34. The apparatus of claim 32 wherein the apparatus includes a means for further dewatering the floccules separated from the water by element (d).

35. The apparatus of claim 34 wherein said means for further dewatering the floccules is a belt filter press.

36. The apparatus of claim 35 wherein means are provided to simultaneously adjust the pH, aerate and agitate the waste water.

37. The apparatus of claim 36 wherein element (d) is a rotary drum filter.

38. The apparatus of claim 33 wherein elements (a), (b), and (c) are integrated into a single unit.

fold A7

08 | 072 418

ABSTRACT OF THE DISCLOSURE

A method for removing metal compounds from waste water comprising the steps of adjusting the pH of the water to from 5 to 12 and preferably 6 to 9; aerating the waste water; adding a flocculating agent to the water and allowing floccules including metal compounds to form; and separating said floccules including metal compounds from the water. An apparatus for carrying out this method is also disclosed.

D024734

EXPRESS MAIL TB208045849US

13

## DECLARATION FOR PATENT APPLICATION

Docket Number: (Optional)

As a below named inventor, I hereby \_\_\_\_\_ re that:

60014-1

My residence, post office address and citizenship are as stated below next to my name.

08 072, 418

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled METHOD AND APPARATUS FOR REMOVING METAL COMPOUNDS FROM WASTE WATER, the specification of which

is attached hereto unless the following box is checked:

was filed on \_\_\_\_\_ as United States Application Number or PCT International Application Number \_\_\_\_\_ and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

## Prior Foreign Application(s)

(Number)	(Country)	(Day/Month/Year Filed)	Priority Claimed
_____	_____	_____	<input type="checkbox"/> Yes <input type="checkbox"/> No
_____	_____	_____	<input type="checkbox"/> Yes <input type="checkbox"/> No
_____	_____	_____	<input type="checkbox"/> Yes <input type="checkbox"/> No

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

(Application Number)	(Filing Date)	(Status - patented, pending, abandoned)
_____	_____	_____

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

DANIEL J. LONG, REGISTRATION NO. 29,404
Address all telephone calls to DANIEL J. LONG at telephone number (412) 471-9633
Address all correspondence to DANIEL J. LONG
2230 KOPPERS BUILDING
436 SEVENTH AVENUE
PITTSBURGH, PA 15219

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor (given name, family name) DALE R. STEVENSON  
 Inventor's signature    Date 5/25/93  
 Residence 124 BRAUN DRIVE, McMURRAY, PA 15317 Citizenship UNITED STATES OF AMERICA  
 Post Office Address 124 BRAUN DRIVE, McMURRAY, PA 15317

Full name of second joint inventor, if any (given name, family name)     
 Second Inventor's signature    Date     
 Residence    Citizenship     
 Post Office Address   

Additional inventors are being named on a separate sheet attached hereto.

D024735

08/07/2418



Applicant SANFORD M. STEVENSON  
Serial or Patent No.: \_\_\_\_\_ Docket No. 60014-1  
Filed or Assigned: CONCURRENTLY HEREWITH Entitled: METHOD AND APPARATUS  
FOR REMOVING METAL COMPOUNDS FROM WASTE WATER  
VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY  
STATUS (37 CFR 1.9(f) AND 1.27(b) - INDEPENDENT INVENTOR

As a below named inventor, I hereby declare that I qualify as an independent inventor as defined in 37 CFR 1.19(c) for purposes of paying reduced fees under section 41(a) and (b) of Title 35, United States Code, to the Patent and Trademark Office with regard to the invention entitled: METHOD AND APPARATUS FOR REMOVING METAL described in

the specification filed herewith  
 application serial no. \_\_\_\_\_, filed \_\_\_\_\_  
 patent no. \_\_\_\_\_, issued \_\_\_\_\_

I have not assigned, granted, conveyed or licensed and am under no obligation under contract or law to assign, grant, convey or license, any rights in the invention to any person who could not be classified as an independent inventor under 37 C.F.R. 1.9(c) if that person had made the invention, or to any concern which would not qualify as a small business concern under 37 C.F.R. 1.9(d) or a non-profit organization under 37 C.F.R. 1.9(e).

Each person, concern or organization to which I have assigned, granted, conveyed, or licensed or am under an obligation under contract or law to assign, grant, convey, or license any rights in the invention is listed below:

no such person, concern, or organization  
 persons, concerns or organizations listed below\*

\*Note: Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. (37 C.F.R. 1.27)

FULL NAME \_\_\_\_\_  
ADDRESS \_\_\_\_\_  
 INDIVIDUAL  SMALL BUSINESS CONCERN  NON-PROFIT ORGANIZATION

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 C.F.R. 1.28(b))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

SANFORD M. STEVENSON  
NAME OF INVENTOR

NAME OF INVENTOR

NAME OF INVENTOR

SANFORD M. STEVENSON  
SIGNATURE OF INVENTOR

NATURE OF INVENTOR

SIGNATURE OF INVENTOR

5/25/83  
DATE

DATE

EXPRESS MAIL TB208045-1

D024736

1. *ORIGINAL* FILED

08 07241

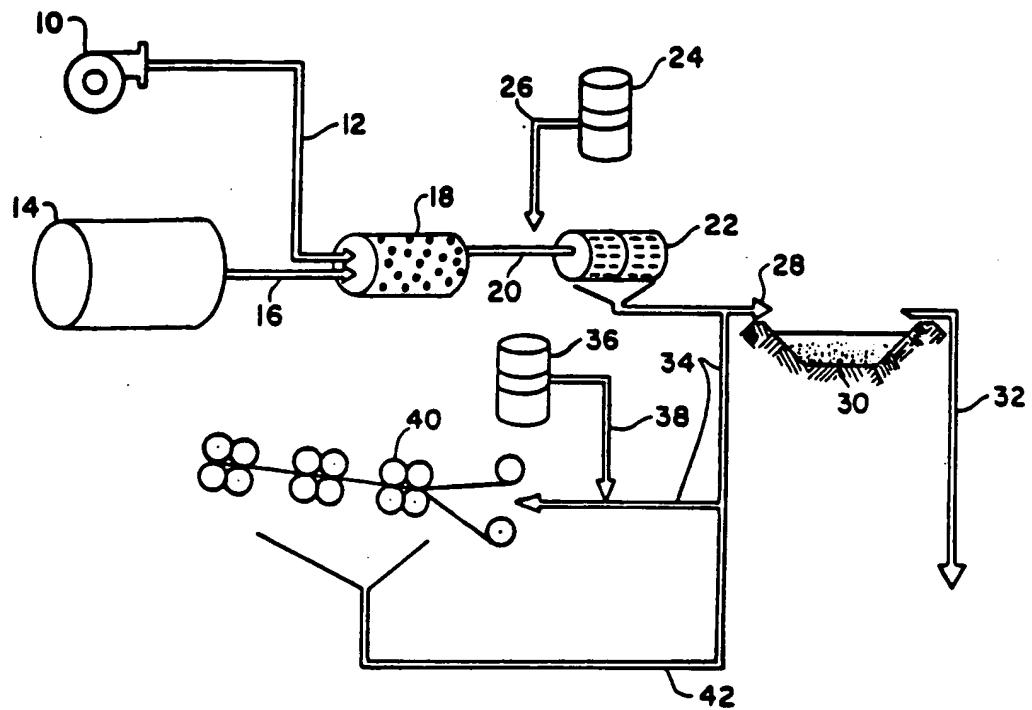


FIG. 1

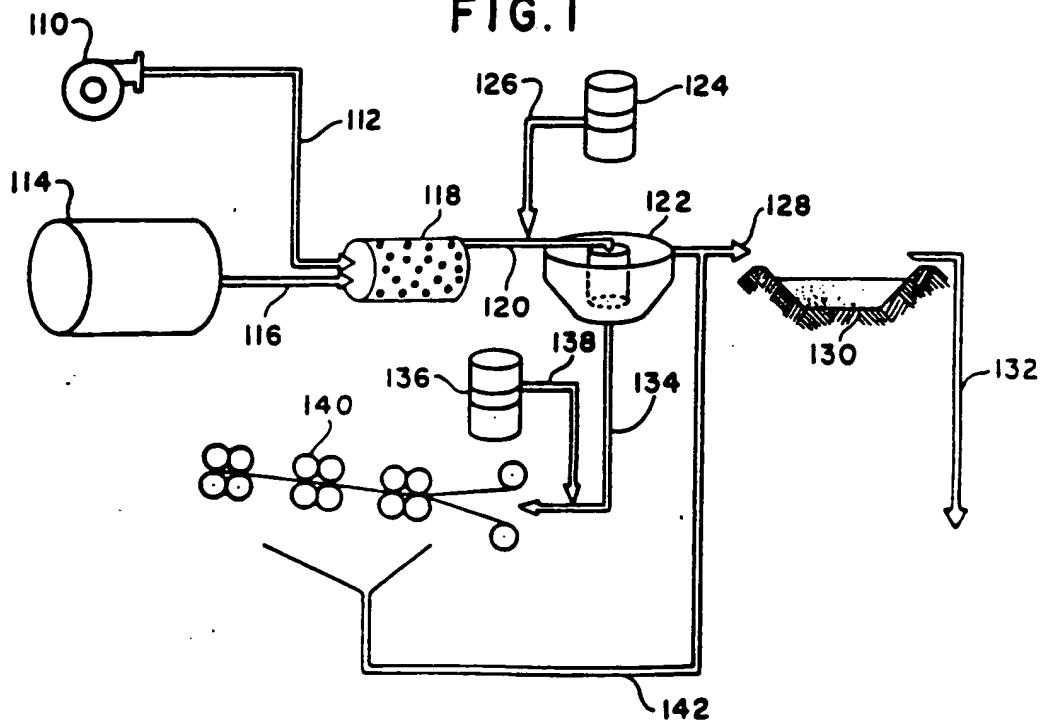


FIG. 2

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SEARCHED MAIL NO. TB208045849US

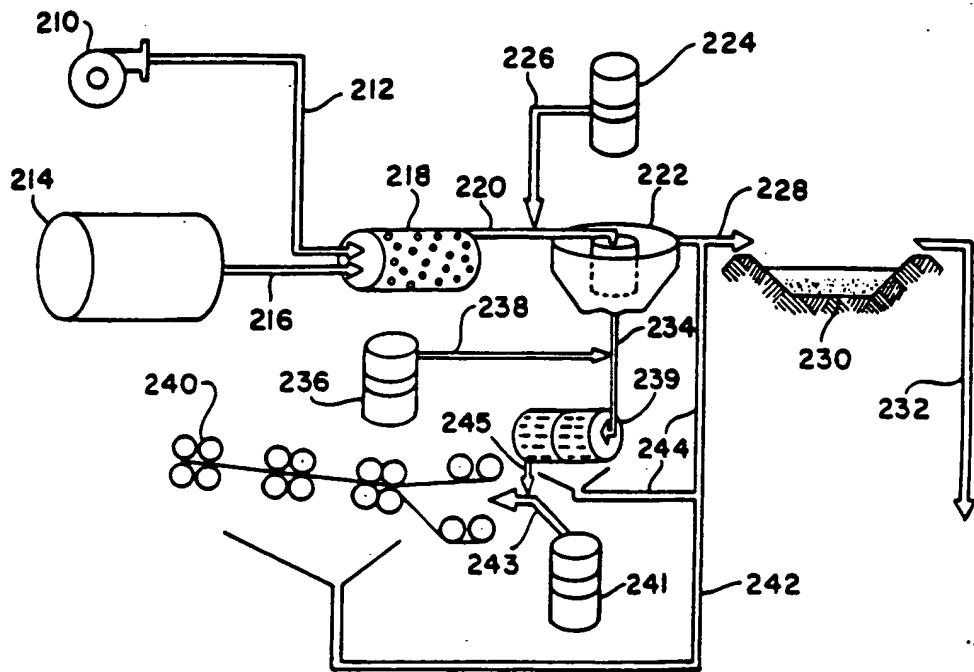


FIG. 3

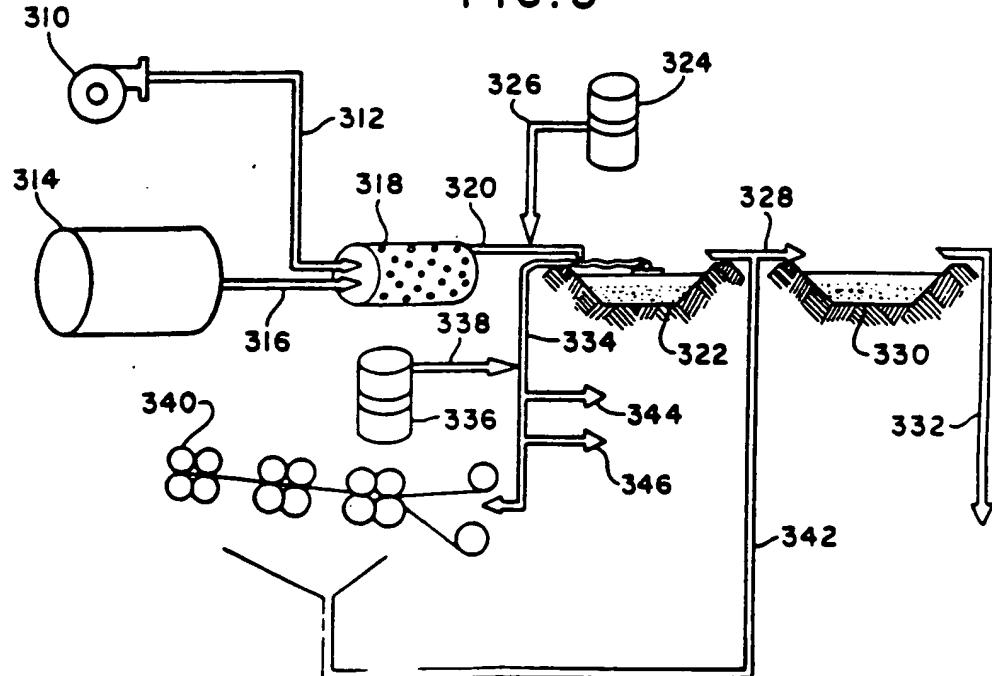


FIG. 4



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Patent and Trademark Office  
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OF PATENTS AND TRADEMARKS  
Washington D.C. 20231

AUG 6 1993

Paper No. 3

In re application of: : DECISION  
Sanford M. Stevenson :  
Serial No. 08/072,418 :  
Filed: May 25, 1993 :  
For: METHOD AND APPARATUS FOR REMOVING :  
METAL COMPOUNDS FROM WASTE WATER :  
PETITION

This is in response to the petition filed May 25, 1993, requesting that the above identified application be granted Special Status under Section 708.02 (VI) of the MPEP.

The petition has been considered and found to be deficient for the following reasons: Section 708.02 (VI) of the MPEP requires that an affidavit/declaration in support of the allegation of the environmental quality enhancement accompany the petition. Since no such affidavit/declaration accompanied the petition, the petition is incomplete. Accordingly, the petition is denied.

*Richard V. Fisher*  
Richard V. Fisher, Director  
Patent Examining Group 1300  
Specialized Chemical Industries  
and Chemical Engineering

Daniel J. Long  
2230 Koppers Building  
436 Seventh Avenue  
Pittsburgh, PA 15219

0024742



UNITED STATES DEPARTMENT OF COMMERCE  
Patent and Trademark Office  
Address: COMMISSIONER OF PATENTS AND TRADEMARKS  
Washington, D.C. 20231

SERIAL NUMBER      FILING DATE      FIRST NAMED INVENTOR      ATTORNEY DOCKET NO.

EXAMINER

ATT. UNIT      PAPER NUMBER

4

04-04-250

This application has been examined       Responsive to communication filed on \_\_\_\_\_  This action is made final.

A shortened statutory period for response to this action is set to expire 3 month(s). \_\_\_\_\_ days from the date of this letter. Failure to respond within the period for response will cause the application to become abandoned. 35 U.S.C. 133

Part I      THE FOLLOWING ATTACHMENT(S) ARE PART OF THIS ACTION:

1.  Notice of References Cited by Examiner, PTO-892.  
2.  Notice of Art Cited by Applicant, PTO-1449.  
3.  Information on How to Effect Drawing Changes, PTO-1474  
4.  Notice re Patent Drawing, PTO-948.  
5.  Notice of Informal Patent Application, Form PTO-152.  
6.  \_\_\_\_\_

Part II      SUMMARY OF ACTION

1.  Claims 1-38 are pending in the application.  
Of the above, claims 3.2-38 are withdrawn from consideration.  
2.  Claims \_\_\_\_\_ have been cancelled.  
3.  Claims \_\_\_\_\_ are allowed.  
4.  Claims 1-31 are rejected.  
5.  Claims \_\_\_\_\_ are objected to.  
6.  Claims 1-38 are subject to restriction or election requirement.  
7.  This application has been filed with informal drawings under 37 C.F.R. 1.85 which are acceptable for examination purposes.  
8.  Formal drawings are required in response to this Office action.  
9.  The corrected or substitute drawings have been received on \_\_\_\_\_. Under 37 C.F.R. 1.84 these drawings are  acceptable,  not acceptable (see explanation or Notice re Patent Drawing, PTO-948).  
10.  The proposed additional or substitute sheets of drawings, filed on \_\_\_\_\_ has (have) been  approved by the examiner,  disapproved by the examiner (see explanation).  
11.  The proposed drawing correction, filed on \_\_\_\_\_ has been  approved,  disapproved (see explanation).  
12.  Acknowledgment is made of the certified copy filed under U.S.C. 119. The certified copy has  been received,  not been received,  been filed in parent application, \_\_\_\_\_; filed on \_\_\_\_\_.  
13.  Since this application appears to be in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under 37 C.F.R. 1.111 and 2.111. A certificate of mailing, 935 O.G. 213.  
14.  Other

EXAMINER'S ACTION

PTOL-329 (Rev. 5-88)

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1. Restriction to one of the following inventions is required under 35 U.S.C. 121:

Group I. Claims 1-31, drawn to a method, classified in Class 210, subclass 721.

Group II. Claims 32-38, drawn to an apparatus, classified in Class 210, subclass 202.

The inventions are distinct, each from the other because of the following reasons:

2. Inventions I and II are related as process and apparatus for its practice. The inventions are distinct if it can be shown that either: (1) the process as claimed can be practiced by another materially different apparatus or by hand, or (2) the apparatus as claimed can be used to practice another and materially different process. (M.P.E.P. § 806.05(e)). In this case the apparatus as claimed can be used to practice a materially different method such as an ore separation method.

3. Because these inventions are distinct for the reasons given above and have acquired a separate status in the art as shown by their different classification and recognized divergent subject matter, restriction for examination purposes as indicated is proper.

4. During a telephone conversation with Daniel J. Long on 11-18-93 a provisional election was made without traverse to prosecute the invention of Group I, claims 1-31. Affirmation of

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this election must be made by applicant in responding to this Office action. Claims 32-38 are withdrawn from further consideration by the Examiner, 37 C.F.R. § 1.142(b), as being drawn to a non-elected invention.

5. The disclosure is objected to because of the following informalities: In the specification on page 3 "velocation" and "ph" should be changed to - relocation - and - pH -, respectively; and on page 4 "125" should be changed to - 126 -. Appropriate correction is required.

6. Claims 2, 18, 22, 26, and 31 are rejected under 35 U.S.C. § 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. In claim 2 step (e) is not in sequence. In claims 18, 22, and 26 "water removed in step (e)" lacks clear antecedent basis. In claim 31 line 3 a comma should be inserted after "lead".

7. The following is a quotation of 35 U.S.C. § 103 which forms the basis for all obviousness rejections set forth in this Office action:

A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

Subject matter developed by another person, which qualifies as prior art or, under subsection (f) or (g) of section 102

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of this title, shall not preclude patentability under this section where the subject matter and the claimed invention were, at the time the invention was made, owned by the same person or subject to an obligation of assignment to the same person.

8. Claims 1, 2, 4-13, 19, 27, 28, and 31 are rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al.. Walker discloses (see cols. 3-5) a method for removing metal compounds from waste water substantially as claimed. The claims differ from Walker by reciting the addition of a flocculating agent to the water to allow floccules including metal compounds to form. Spence et al. discloses (see cols. 2 and 3) that it is known in the art to add cationic coagulating agents and anionic flocculating agents to form a floc and facilitate removal of heavy metals from waste water. It would have been obvious to one skilled in the art to modify the method of Walker by addition of the cationic coagulating agents and anionic flocculating agents taught in Spence et al., to form a floc and facilitate the removal of heavy metals from the waste water. With regard to claims 10-13, it well known in the art of waste water treatment to utilize the polymers of Spence et al. for dewatering, clarification, and settling purposes, and to add these polymers as dilute aqueous solutions.

9. Claims 3, 16, 17, 20, and 21 are rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al. and further in view of Taylor et al.. The claims differ Taylor

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et al. by reciting that the floccules are further dewatered by the addition of additional flocculating agent and by using a belt filter press. Taylor et al. disclose (see cols. 4, 11, and 12) that it is known in the art to dewater heavy metal residues by the addition of flocculating agents and by using a belt filter press. It would have been obvious to one skilled in the art to modify the references as applied above, by addition of additional flocculating agent and by use of a belt filter press in view of Taylor et al., to aid in dewatering of the floccules.

10. Claim 14 is rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al., as above and further in view of Whittle. The claim differs from the references as applied above by reciting that a portion of the separated water is removed to a polishing pond. Whittle discloses (see Fig. 1 and col. 6) that it is known in the art of water treatment to transfer waste water to a polishing pond for final treatment before being discharged into a receiving stream. It would have been obvious to one skilled in the art to modify the references as applied above, by utilizing a polishing pond in view of the teachings of Whittle, to further treat the waste water prior to discharge into a receiving stream.

11. Claims 15, 23, 24, 29, and 30 are rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al. as above, and further in view of Bastgen et al.. The claims

differ from the references as applied above by reciting that the floccules are separated in a rotary drum thickener. Bastgen et al. discloses (see cols. 5 and 6) that it is known in the art of water treatment to utilize a rotary drum thickener to thicken and dewater flocculated solids. It would have been obvious to one skilled in the art to modify the references as applied above, by utilizing a rotary drum thickener in view of Bastgen et al., to aid in separating the floccules from the water. The use of a specific sequence of separation and additional flocculating agent, would have been an obvious matter of process optimization to one skilled in the art, depending on the specific waste water treated and results desired, absent a sufficient showing of unexpected results.

12. Claims 18 and 22 are rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al. and Taylor et al. as above and further in view of Whittle. The claims differ from the references as applied above by reciting that a portion of the separated water is removed to a polishing pond. Whittle discloses (see Fig. 1 and col. 6) that it is known in the art of water treatment to transfer waste water to a polishing pond for final treatment before being discharged into a receiving stream. It would have been obvious to one skilled in the art to modify the references as applied above, by utilizing a polishing pond in

view of the teachings of Whittle, to further treat the waste water prior to discharge into a receiving stream.

13. Claim 25 is rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al. and Taylor et al. and further in view of Bastgen et al.. The claim differs from the references as applied above by reciting that the floccules are separated in a rotary drum thickener. Bastgen et al. discloses (see cols. 5 and 6) that it is known in the art of water treatment to utilize a rotary drum thickener to thicken and dewater flocculated solids. It would have been obvious to one skilled in the art to modify the references as applied above, by utilizing a rotary drum thickener in view of Bastgen et al., to aid in separating the floccules from the water.

14. Claim 26 is rejected under 35 U.S.C. § 103 as being unpatentable over Walker in view of Spence et al., Taylor et al., and Bastgen et al. as above, and further in view of Whittle. The claim differs from the references as applied above by reciting that the separated water is removed to a polishing pond. Whittle discloses (see Fig. 1 and col. 6) that it is known in the art of water treatment to transfer waste water to a polishing pond for final treatment before being discharged into a receiving stream. It would have been obvious to one skilled in the art to modify the references as applied above, by utilizing a polishing pond in

Serial Number: 08/072,418

-8-

Art Unit: 1308

view of the teachings of Whittle, to further treat the waste water prior to discharge into a receiving stream.

15. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure.

16. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Peter A. Hruskoci whose telephone number is (703) 308-3839. Any inquiry of a general nature or relating to the status of this application should be directed to the Group receptionist whose telephone number is (703) 308-0651.

pah  
November 23, 1993

*Peter A. Hruskoci*  
Peter A. Hruskoci  
Primary Examiner  
Art Unit 1308

D024750

#8/B

PAH

6/17/94

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Sanford M. Stevenson

Serial No. 08/072,418

Examiner: P. Hruskoci

Filed: May 25, 1993

Group Art Unit: 1308

Entitled: **METHOD AND APPARATUS FOR REMOVING  
METAL COMPOUNDS FROM WASTE WATER**

**OFFICIAL**

- - -000- -

Pittsburgh, Pennsylvania 15219  
June 17, 1994

Non. Commissioner of Patents and Trademarks  
Washington, D.C. 20231

AMENDMENT

Sir:

Please amend the above captioned application as  
follows:

IN THE SPECIFICATION:

On page 3, line 15, after "about", delete ".10" and  
substitute ---.01---.

IN THE CLAIMS:

*Att  
6/17/94 C*  
B1  
1. (Twice Amended) A method for removing metal  
compounds selected from iron, manganese, aluminum, zinc, copper,  
lead, arsenic and chromium from waste water comprising the steps  
of:

- (a) adjusting the pH of the waste water to from about  
5 to about 12;
- (b) aerating the waste water;

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*11/19/94 C*

- (c) [simultaneously with step (b)] agitating the waste water, wherein steps (a), (b) and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration of from about .01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a metals concentration of from about 50 mg./l. to about 1,000 mg./l.
- (d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said metal compounds to form; and
- (e) then separating said floccules including said metal compounds from the water.

Cancel claims 5, 31, and 39.

REMARKS

*Interview  
Record OK  
GAR  
6/17/94*

Reconsideration is respectfully request. The above amendment is made pursuant to a telephone interview conducted June 16, 1994 with the Examiner. Support for the amendment to the specification is at page 2, line 1. The use of the number ".10" rather than ".01" at page 3, line 15 was due to a clerical error.

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WILSON, ROBERT L. *How will he fit in?* 1966

i 444

The applicant wishes to make of record that the undersigned attorney and the Examiner agreed during their telephone interview that the purpose of the aeration and flow rate limitations added by the amendment to step (c) of claim 1 was to define a range of dissolved oxygen concentrations for the metals concentrations which would result from the recited ratios of aeration and flow rate limitations. The intention of this language was not to limit the application of this claim to any particular waste water flow rate such as 50 gal./min. to 500 gal./min.

In view of the foregoing, the instant application is believed to be in condition for allowance, and, therefore, an early issuance thereof is earnestly solicited.

If the Examiner believes that a telephone interview would be beneficial to advance prosecution of the instant application to early issue, he is invited to contact the undersigned at the telephone number listed below.

Respectfully submitted,

David J. Long

Daniel J. Long  
Attorney for Applicant

DJLong/lcl  
Area Code 412-471-9633  
FAX 412-765-1583

CERTIFICATION OF FACSIMILE TRANSMISSION  
I hereby certify that this paper is being facsimile  
transmitted to the Patent and Trademark Office on the date shown  
below and to facsimile number 703-305-3601.

Linda C. LaCone  
Linda C. LaCone

June 17, 1994

1

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0024856



UNITED STATES DEPARTMENT OF COMMERCE  
Patent and Trademark Office

Address: Box ISSUE FEE  
COMMISSIONER OF PATENTS AND TRADEMARKS  
Washington, D.C. 20231

D3M1/0623

DANIEL J. LONG  
2230 KOPPERS BUILDING  
436 SEVENTH AVENUE  
PITTSBURGH, PA 15219

NOTICE OF ALLOWANCE  
AND ISSUE FEE DUE

Note attached communication from the Examiner  
 This notice is issued in view of applicant's communication filed \_\_\_\_\_

SERIES CODE/SERIAL NO.	FILING DATE	TOTAL CLAIMS	EXAMINER AND GROUP ART UNIT	DATE MAILED
08/072,418	05/25/93	025	HRUSKOCI, P	1308 06/23/94

First Named  
Applicant

STEVENSON

SANFORD M.

TITLE OF  
INVENTION

METHOD FOR REMOVING METAL COMPOUNDS FROM WASTE WATER  
(AS AMENDED)

	ATTY'S DOCKET NO.	CLASS-SUBCLASS	BATCH NO.	APPLN. TYPE	SMALL ENTITY	FEES DUE	DATE DUE
1	600141	210-710.000	R91	UTILITY	YES	\$585.00	09/23/94

THE FEE DUE IS THE AMOUNT IN EFFECT AT THIS TIME. IF THE AMOUNT OF THE ISSUE FEE INCREASES PRIOR TO PAYMENT, APPLICANT WILL BE NOTIFIED OF THE BALANCE OF ISSUE FEE DUE.

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT.

PROSECUTION ON THE MERITS IS CLOSED.

THE ISSUE FEE MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED.

HOW TO RESPOND TO THIS NOTICE:

I. Review the SMALL ENTITY Status shown above.  
If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status:

A. If the status is changed, pay twice the amount of the FEE DUE shown above and notify the Patent and Trademark Office of the change in status, or  
B. If the Status is the same, pay the FEE DUE shown above.

II. Part B of this notice should be completed and returned to the Patent and Trademark Office (PTO) with your ISSUE FEE. Even if the ISSUE FEE has already been paid by charge to deposit account, Part B should be completed and returned.

III. If communications regarding this application must give series code (or filing date) and serial number. If communications prior to issuance to Box ISSUE FEE unless advised to contrary.

**NOTE TO APPLICANT:** Patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

PTOL-68 (REV.7-88) (OMB Clearance is pending)

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UNITED STATES DEPARTMENT OF COMMERCE  
Patent and Trademark Office  
Address: COMMISSIONER OF PATENTS AND TRADEMARKS  
Washington, D.C. 20231

SERIAL NUMBER	08/072,418	FILED DATE	05/25/93	FIRST NAMED APPLICANT	STEVENSON	ATTORNEY DOCKET NO.	S 600141
				D3M1/0623		HRUSKOCIL, P. EXAMINER	
						ART UNIT	PAPER NUMBER
						1308	8 1/2

DANIEL J. LONG  
2230 KOPPERS BUILDING  
436 SEVENTH AVENUE  
PITTSBURGH, PA 15219

DATE MAILED: 06/23/94

EXAMINER INTERVIEW SUMMARY RECORD

All participants (applicant, applicant's representative, PTO personnel):

(1) Peter R. Hruskocil (3) \_\_\_\_\_  
(2) \_\_\_\_\_ (4) \_\_\_\_\_

Date of interview 6-21-94

Type:  Telephonic  Personal (copy is given to  applicant  applicant's representative).

Exhibit shown or demonstration conducted:  Yes  No. If yes, brief description: \_\_\_\_\_

Agreement  was reached with respect to some or all of the claims in question.  was not reached.

Claims discussed: 1, 18, 19 & 29

Identification of prior art discussed: None

Description of the general nature of what was agreed to if an agreement was reached, or any other comments: It was agreed to amend the claims as shown in the interview. Amendment for the sake of clarity & accuracy.

(A fuller description, if necessary, and a copy of the amendments, if available, which the examiner agreed would render the claims allowable must be attached. Also, where no copy of the amendments which would render the claims allowable is available, a summary thereof must be attached.)

Unless the paragraphs below have been checked to indicate to the contrary, A FORMAL WRITTEN RESPONSE TO THE LAST OFFICE ACTION IS NOT WAIVED AND MUST INCLUDE THE SUBSTANCE OF THE INTERVIEW (e.g., items 1-7 on the reverse side of this form). If a response to the last Office action has already been filed, then applicant is given one month from this interview date to provide a statement of the substance of the interview.

It is the responsibility of the applicant to provide a response record of the substance of the interview.

The examiner's interview summary above (including any attachments) reflects a complete response to each of the objections, rejections and other actions that may be present in the last Office action and since the claims are now allowable, this completed form is considered to fulfill the requirements of the last Office action.

Peter R. Hruskocil  
Examiner's Signature

PTO-413 (REV. 1-90)

ORIGINAL FOR INS.

10 HAND FLAP OF FILE WRAPPER

0024859



UNITED STATES DEPARTMENT OF COMMERCE

Patent and Trademark Office

Address: COMMISSIONER OF PATENTS AND TRADEMARKS  
Washington, D.C. 20231

SERIAL NUMBER	FILING DATE	FIRST NAMED APPLICANT	ATTORNEY DOCKET NO.
09/072,418	05/25/93	STEWARTSON DANIEL J. LONG	HRUSKOCIA
		D3M1/0623	
2230 KOPPERS BUILDING 436 SEVENTH AVENUE PITTSBURGH, PA 15219			EXAMINER 1308
			ART UNIT 66 CARRY NUMBER 81/2
DATE MAILED:			

## EXAMINER INTERVIEW SUMMARY RECORD

All participants (applicant, applicant's representative, PTO personnel):

(1) Mr. Long (3) \_\_\_\_\_

(2) \_\_\_\_\_ (4) \_\_\_\_\_

Date of interview 6-16-94Type:  Telephonic  Personal (copy is given to  applicant  applicant's representative).Exhibit shown or demonstration conducted:  Yes  No. If yes, brief description: \_\_\_\_\_Agreement  was reached with respect to some or all of the claims in question.  was not reached.Claims discussed: AllIdentification of prior art discussed: Free list of prior art

Description of the general nature of what was agreed to if an agreement was reached, or any other comments: Amendments to the claims were discussed to patentably distinguish over the prior art of record. Mr. Long agreed to FAX as supplemental amendment for formal consideration.

(A fuller description, if necessary, and a copy of the amendments, if available, which the examiner agreed would render the claims allowable must be attached. Also, where no copy of the amendments which would render the claims allowable is available, a summary thereof must be attached.)

Unless the paragraphs below have been checked to indicate ... the comments, A FORMAL WRITTEN RESPONSE TO THE LAST OFFICE ACTION IS NOT WAIVED AND MUST INCLUDE THE SUBSTANCE OF THE INTERVIEW (e.g., items 1-7 on the reverse side of this form). If a response to the last Office action has already been filed, then applicant is given one month from this interview date to provide a statement of the substance of the interview which will be considered a response to this Office action.

... the interview summary above, including any amendments, reflects a complete response to each of the objections, rejections and notices of non-compliance that may be present in the last Office action, and where the claims are now allowable, this completed form is considered to fulfill the requirements of the last Office action.

Peter A. Hruskocia  
Examiner's Signature

PTOL-413 (REV. 1-93)

ORIGINAL FOR INVENTOR - ATTACH TO HAND FLAP OF FILE WRAPPER

D024860



UNITED STATES DEPARTMENT OF COMMERCE  
Patent and Trademark Office  
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Washington, D.C. 20231

SERIAL NUMBER	FILING DATE	FIRST NAMED APPLICANT	ATTORNEY DOCKET NO.
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08/072,418 05/25/93 STEVENSON

S EXAMINER 141

DANIEL J. LONG  
2230 KOPPERS BUILDING  
436 SEVENTH AVENUE  
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D3M1/0623

HRUSKOC, P

ART UNIT PAPER NUMBER

#91C

DATE MAILED 6/21/94

Entered Part  
6/21/94

### NOTICE OF ALLOWABILITY

#### PART I.

- This communication is responsive to the paper was filed 5-9-6-17-94.
- All the claims being allowable, PROSECUTION ON THE MERITS OF (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice Of Allowance And Issue Fee Due or other appropriate communication will be sent in due course.
- The allowed claims are 1, 3, 4, 6, & 10-30.
- The drawings filed on \_\_\_\_\_ are acceptable.
- Acknowledgment is made of the claim for priority under 35 U.S.C. 119. The certified copy has [...] been received. [...] not been received. [...] been filed in parent application Serial No. \_\_\_\_\_, filed on \_\_\_\_\_.
- Note the attached Examiner's Amendment.
- Note the attached Examiner Interview Summary Record, PTO-413. (2)
- Note the attached Examiner's Statement of Reasons for Allowance.
- Note the attached NOTICE OF REFERENCES CITED, PTO-892.
- Note the attached INFORMATION DISCLOSURE CITATION, PTO-1449.

#### PART II.

A SHORTENED STATUTORY PERIOD FOR RESPONSE to comply with the requirements noted below is set to EXPIRE THREE MONTHS FROM THE "DATE MAILED" indicated on this form. Failure to timely comply will result in the ABANDONMENT of this application. Extensions of time may be obtained under the provisions of 37 CFR 1.136(a).

- Note the attached EXAMINER'S AMENDMENT or NOTICE OF INFORMAL APPLICATION, PTO-152, which discloses that the oath or declaration is deficient. A SUBSTITUTE OATH OR DECLARATION IS REQUIRED.
- APPLICANT MUST MAKE THE DRAWING CHANGES INDICATED BELOW IN THE MANNER SET FORTH ON THE REVERSE SIDE OF THIS PAPER.
  - Drawing informities are indicated on the NOTICE RE PATENT DRAWINGS, PTO-948, attached hereto or to Paper No. \_\_\_\_\_, CORRECTION IS REQUIRED.
  - The proposed drawing correction filed on \_\_\_\_\_ has been approved by the examiner. CORRECTION IS REQUIRED.
  - Approved drawing corrections are described by the examiner in the attached EXAMINER'S AMENDMENT. CORRECTION IS REQUIRED.
  - Formal drawings are now REQUIRED.

This letter should include in the upper right hand corner, the following information from the NOTICE OF ALLOWABILITY: FEE DUE, ISSUE BATCH NUMBER, DATE OF THE NOTICE OF ALLOWANCE, AND SERIAL NUMBER.

- Examiner Interview Summary Record, PTO-413 (2)  
- Reasons for Allowance  
- Notice of References Cited, PTO-892  
X Information Disclosure Citation, PTO-1449

Notice of Informal Application, PTO-152  
Notice re Patent Drawings, PTO-948  
Listing of Bonded Draftsmen  
Other

D024861

IN THE UNITED STATES COURT OF FEDERAL CLAIMS

CHEMICAL SEPARATION TECHNOLOGY, INC., )  
and SANFORD M. STEVENSON, )  
Plaintiffs, ) No. 97-21C  
v. )  
THE UNITED STATES, ) Judge Francis M. Allegra  
Defendant. )

DECLARATION OF JOHN A. ROTH

John A. Roth, of Nashville, Tennessee, declares under penalty of perjury that the following is true and correct:

1. I am a registered professional engineer in the state of Kentucky, have a bachelors (1956) and masters (1957) in chemical engineering from the University of Louisville, and a Ph.D. (1961) in chemical engineering from University of Louisville. I served two years in the Airforce, and have been an assistant professor to professor at Vanderbilt University from 1962 to the present, teaching chemical and environmental engineering. In 1976-77, I served as the commissioner for environmental protection for the Commonwealth of Kentucky. I have been active in environmental consulting and research from 1972 to the present. My specific consulting and research projects, as well as publications are set forth in my C.V. attached hereto as Exhibit 1. With regard to waste water treatment chemistry and technology my specific experience would include industrial waste water treatment with emphasis in chemical oxidation. I have analyzed waste water treatment systems, and am familiar with the techniques, apparatus,

operations, and chemistry utilized in the waste water treatment industry.

2. I have been asked by Chemical Separation Technology, Inc., and Sanford M. Stevenson, to provide my opinions regarding the scope and validity of U.S. Patents Nos. 4,749,497 and 5,370,800 in connection with their claim of infringement by the United States Government.

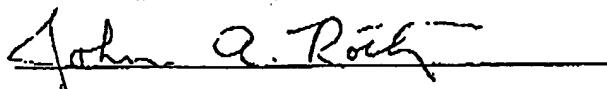
3. Within the preceding four years I have not testified as an expert at trial or by deposition.

4. I am charging \$150.00 (ONE HUNDRED AND FIFTY DOLLARS) per hour plus expenses for preparing the attached report and providing testimony at trial in November of this year.

5. Attached to this declaration is a true and correct copy of my report, containing my opinions, all of which are based my experience and the references attached, as well as all the documents identified by Mr. Dupon's report, and Mr. Dupon's report, and are expressed within a reasonable degree of chemical and environmental engineering certainty. (Report attached as Exhibit 2)

6. I declare under penalty of perjury that this declaration is true and correct.

Dated: June 22, 2000

  
John A. Roth

**John A. Roth**  
**Department of Chemical Engineering**  
**Box 1604, Sta. B**  
**Vanderbilt University**  
**Nashville, Tennessee 37235**  
**June 22, 2000**

Louis M. Tarasi, Jr., Esquire  
The Tarasi Law Firm, P.C.  
510 Third Ave.  
Pittsburgh, PA 15219

You have asked me to express an expert opinion with respect to CST's and Mr. Stevenson's Patents. As an introduction to my opinion with regard to the 497 and 800 Patents of CST and Mr. Stevenson, it is clear from the history of the wastewater treatment industry that CST and Mr. Stevenson developed patents to have a contained portable system of treating mine wastewater. These systems were such that they could be placed in trailers and utilized to treat water which was previously treated by using ponds and other methods which required a great deal of land and a great deal of time. Because of this improved process, wastewater could be treated effectively to the point where the water would be usable and recyclable because the metals in the wastewater were precipitated and removed quite quickly and effectively from the water. In my opinion, both patents reflect something new and novel in the wastewater treatment industry. The obvious fact is that the history of the industry shows that nothing like this was on the scene or in use in any form whatsoever before the inventions of CST and Stevenson.

Therefore, the patented apparatus and methods set forth by the 497 patent owned by CST and the 800 patent owned by Mr. Sanford Stevenson represent a significant improvement over the prior art. I have reviewed the patents involved, their prosecution histories, operational data, the opinion of Mr. Dupon, and the documents submitted with his report, and as a result, and in light of this factual background render my opinion as follows:

As an initial matter, with reference to the technology disclosed in the 497 and 800 patents, I would define one skilled in the art as a person with an accredited bachelors degree, (Accreditation Board for Engineering and Technology, Inc., (ABET) Accredited Degree) in the areas of either environmental, chemical, civil, or mining engineering and about four years of responsible charge in design, research, development, testing or evaluation of industrial waste water treatment systems.

An individual with this background would be able to read the patents and build and operate the inventions disclosed in the patents, because he would appreciate the chemistry, and would be able to design, construct and operate the systems to achieve the results which are the objects of the patents disclosed.

### Mine Wastewater Chemistry

Mr. Dupon contends that “the pH of water is a measure of hydrogen ion activity, and represents an indirect indication of its acidity and alkalinity”. He further contends, that “Generally speaking, the higher the pH, the more alkaline the water, the lower the pH, the more acidic it is, while a pH of 7 indicates neutrality”. (Pg. 4, Dupon Report) This is an incorrect and misleading statement. In fact,

$$\text{Acidity} = 2[\text{H}_2\text{CO}_3] + [\text{HCO}_3^-] + [\text{H}^+] - [\text{OH}^-] + \sum n\text{Me}^{+n}$$

where,  $\text{Me}^{+n}$  represents an amphoteric metal (such as  $\text{Fe}^{+2}$ ) in solution. Therefore, in a case of acid mine drainage, it is the metals which make up the bulk of acidity, and the hydrogen ion activity is a minor component of acidity.<sup>1</sup>

### Treatment Techniques

Citing the Environmental Protection Agency’s Design Manual: Neutralization of Acid Mine Drainage, Mr. Dupon contends that the use of a single reactor for oxidation and neutralization of acid mine drainage was already accomplished. He refers to Figures 2-3 and 7-5, in the EPA Manual, both of which clearly show a recycle of sludge. The increased volume of reactor required and increased sludge handling requirements with a recycle stream of precipitated solids is quite different than those in the patent. The manner in which influent (raw wastewater), neutralizing agent (i.e., caustic), and oxygen are contacted with additional agitation in a relatively small tank is quite different from any process suggested in the EPA Manual. Accordingly, the results obtained using a relatively short residence time are not anticipated or obvious in light of the EPA Manual or the patents to which it refer.

### Definition of Claim Terms

Having read the claims of the 800 and 497 patents, the written descriptions and figures, the Patent Office files of both patents, and Mr. Dupon’s analysis, I disagree with Mr. Dupon’s claim interpretation on the following points:

### 800 Patent Claims

This Patent disclosed its object and scope in Col. 1, lines 5-22, under Background of the invention. In this section, the patent discloses that the invention relates to the treatment of “waste water, and in particular to the treatment of water to remove various precipitated or suspended metal compounds therefrom.” The Background also relates that prior treatment techniques have been unsatisfactory because of “the lengthy periods of time which they would generally be required to effect treatment.”

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<sup>1</sup>Vernon L. Snoeyink and David Jenkins, 1980, Water Chemistry, John Wylie & Sons, Inc.

I understand, therefore, that the present invention is intended to remove metal contaminants from a waste stream in a shorter residence time than was experienced prior to May 25, 1993, the date of application for the patent.

As an initial matter, Mr. Dupon seems to treat even the clearest of terms in the patents as requiring some technical meaning beyond their ordinary meaning, in light of the object and scope of the patent.

For instance, Mr. Dupon contends that the term "removing" (Col. 3, Ln. 57) is imprecise, as it does not suggest a reduction of a particular amount. Nothing in the language of the patent's claims addresses reduction of metals to a particular amount. Remove by definition means "to take away, extract, do away with" <sup>2</sup>. In my opinion the general definition of "remove" is consistent with the object and claims of the patent, *i.e.*, the removal of metals from a waste stream, and no technical interpretation is required for the term "remove", nor would one skilled in the art impose the meaning Mr. Dupon does upon that term. Indeed to define the term as Mr. Dupon does, is to impress the invention with a character that does not read consistently with the object and scope of the patented invention.

Mr. Dupon's analysis of the meaning of "metal compounds" and "selected from iron, manganese, aluminum, zinc, copper, lead, arsenic, and chromium" (Col. 5, Ln. 57, & Col. 3, Lns. 58-59) is incorrect. In my opinion, one skilled in the art would read "metal compounds" to refer to metal oxyhydroxides existing either in solution or as precipitates.

Significant concentrations of chromium and/or manganese may be present in these wastewaters. Chromium exists in aqueous solution primarily as the trivalent Cr<sup>+3</sup> when Fe<sup>+2</sup> is present since it serves as a reducing agent for Cr(VI). With the addition of oxygen, Cr<sup>+3</sup> remains in the trivalent form and upon neutralization precipitates out as the hydroxide precipitate, Cr(OH)<sub>3</sub> (s). While it is not necessary to aerate the solution for Cr<sup>+3</sup> or other non-reactive species, such as Al<sup>+3</sup>, the aeration will not hinder the removal in light of the evidence that Fe(OH)<sub>3</sub> is demonstrated to be removed with the application of aeration. Copper may have some redox behavior and aeration may be required for its removal. Prior to actually removing copper from a waste stream with the method and apparatus of the 800 patent, however, one skilled in the art would not be able to predict with confidence that the invention would remove copper.

Mr. Dupon's reads claim 1(a) "adjusting the pH of the waste water to from about 5 to about 12" (Col. 3, Lns. 61-62) to mean that the pH of the waste water must start out outside the range of from about 5 to about 12. Mr. Dupon also states that the term "about" is imprecise and would not enable one skilled in the art to determine which pH to use. Notably, on page 5 and 6 of his report, Mr. Dupon, in describing basic Mine Wastewater Chemistry uses the same expression. He states "Below a pH of about 4 to 5" and "very slowly unless the pH exceeds about 6". It would seem to me that Mr. Dupon, who claims to be one skilled in the art, understands his use of "about" and should also understand the claims' use of "about". One

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<sup>2</sup>American Heritage Dictionary (2<sup>nd</sup> Ed.) 1988, pg. 583

skilled in the art would understand that an increase of 1 pH unit represents an exponential (10 times) increase in concentration.

I do not believe that a person skilled in the art would impose the meaning Mr. Dupon has that the pH of the water must start out outside the range of 5 to 12. An adjustment can occur from 6 to 7 or 3 to 12 and still be understood to mean an adjustment from about 5 to about 12. I would think one skilled in the art would understand that to accomplish the initial precipitation of the metals from the waste stream, a target pH of from about 5 to about 12 would be selected, depending upon the metal of concern. Because of the wide variability of different wastes, the best operating pH must be selected by experimentation. This is clear to anyone in this field that every metal has its own pH of minimum solubility and in complex wastes there are often unpredictable interactions.

With regard to the 800 Patent I would not agree with Mr. Dupon that the phrase "Aerated in said reaction tank to provide a dissolved oxygen concentration from about 0.01 lbs/hr to about 70 lbs/hr" is ambiguous. Mr. Dupon failed to read the entire claim, which shows the flow rate in conjunction with the rate of aeration. Accordingly, the phrase relates to the rate of aeration. The example set forth in the patent that provides for a "dissolved oxygen concentration of 3 lbs/HP hr." with a corresponding flow rate is clearly consistent with the claim language. (Col. 3, Lns 31-32). Therefore, one skilled in the art would conclude that the range relates to the rate of aeration, that will supply the needed oxygen concentration to effect an appropriate reaction based upon the influent flow rate.

One skilled in the art, therefore, could easily calculate the equivalent oxygen concentration based upon the rate of aeration by using the following formula:

$$\text{Equivalent Oxygen Concentration} = \text{rate} \times \text{power/influent flow rate}$$

Or, for a given rate of 3lb/HP-hr and using a 7.5 HP aerator motor and influent flow rate of 450 gal/min. the equivalent oxygen concentration is 99.8 mg/l.

Indeed Mr. Dupon in paragraph 32 of his report recognizes, as much when he concludes that support is found within the patent to conclude that the rate of aeration is what is intended to be disclosed and not the oxygen concentration. Accordingly, one skilled in the art would conclude that the rate of aeration "from between 0.01 lbs/hr to about 70lbs/hr" would provide the appropriate oxygen concentration to effect the reaction desired.

I agree with Mr. Dupon that "Floccule" is not a standard term of art used in this industry. The term, however, is defined as "a soft mass of matter resembling a soft tuft of wool" according to Webster's New World Dictionary, College Edition. So defined, the meaning is consistent with the object of the patent which seeks to solidify the metal wastes into a sludge for handling and disposal. Metal Hydroxide sludges are "soft masses of matter".

With regard to polymer dilution concentrations, past practice and subsequent adjustment for specific wastes based upon experimentation determine the dilution used. To one skilled in the art, experimentation with polymers would be expected.

In short, in my opinion, I think one skilled in the art would interpret the above terms consistently with the object of the patent, that is, to remove metal compounds from waste streams using a lower retention time as compared with the prior art.

#### 497 Claims

In paragraph 49 of his report, Mr. Dupon contends that "treatment" as used in the claim is ambiguous. One skilled in the art would understand treatment as used in the patent to refer to the "treatment" of water to effect the desired result, namely, "for the purpose of precipitating metals and neutralization of acid found in waters such as that which results from mine drainage." (Col. 1 &2, Lns. 68; 1-2). Mr. Dupon's contention that the term would be ambiguous to one skilled in the art is simply not true. One skilled in the art would understand "treatment" in the context of the patent to refer to the object of the invention as disclosed.

One skilled in the art would understand the language, "Automated metering and control means", in the context of this patent using its unique design, to mean control the pH in the entire reactor. This is effected using adequate agitation and a pH probe and a metering pump in the control loop, which one skilled in the art would understand implicitly.

"Introducing a source of acidic or metal bearing water into a treatment unit" means feeding the raw wastewater (influent) into the reactor.

I have examined drawings of the several attempted modifications of the 497 patent by the defendants. In my opinion, if any of these show an apparatus which is outside of the literal scope of the 497 patent, they still perform substantially the same function, in substantially the same way, to achieve substantially the same results as the elements recited in the claims of the 497 patent.

#### Invalidity Issues

The patents are not indefinite in light of their claims. For instance, the patent is self evident as to the meaning of remove. Mr. Dupon's reads claim 1(a) "adjusting the pH of the waste water to from about 5 to about 12" (Col. 3, Lns. 61-62) to mean that the pH of the waste water must start out outside the range of from about 5 to about 12. Mr. Dupon also states that the term "about" is imprecise and would not enable one skilled in the art to determine which pH to use. Notably, on page 5 and 6 of his report, Mr. Dupon, in describing basic Mine Wastewater Chemistry uses the same expression. He states "Below a pH of about 4 to 5" and "very slowly unless the pH exceeds about 6". This same concept applies to most of these parameters given the wide differences in wastes and even variability within a single waste stream. It is clear, therefore, to one skilled in the art that this process dealing with various metals has to have a range of pH values and it does.

**Enablement 800 Patent**

It is my opinion that one skilled in the art is able to design, construct, and operate a treatment system based on the claims, drawings, and specifications set forth in this patent.

**Enablement 497 Patent**

The description of the contacting of raw wastewater (influent), neutralizing agent (i.e., caustic), and air; the agitation; and the uniform pH control throughout the reactor results in the neutralization, oxidation, and precipitation of flocs in shorter than previously used residence times, results in a high quality floc. This compact, relatively small reactor achieves the stated goals of the Patent. It is my opinion that one skilled in the art is able to design, construct, and operate a treatment system based on the claims, drawings, and specifications set forth in this patent.

**Anticipation and Obviousness - EPA Manual**

The chemistry associated with removal of metal compounds from acid mine drainage is standard. The process, techniques and configuration of the 497 reactor and process variables, *viz.* residence times and oxygen mass transfer rates, as further articulated in the 800 patent, however, are unexpected and surprising in light of the prior art identified by Mr. Dupon. The process configuration results in virtual instantaneous uniform pH within the reactor producing flocs for which the resulting dewatering is much simpler, faster and more cost effective.

Mr. Dupon, on page 38 of his report, states that the configuration of the 497 reactor is “probably not capable of increasing the oxidation rate without use of oxidants other than air. As explained above, and in the EPA Manual, oxidation by aeration is typically more complete when it does not occur at roughly the same point as addition of pH adjustment chemical, since the oxygen added is intended to replace oxygen depleted by the oxidation of metals.”

While this statement is true, generally, it is not true in light of what is claimed in both patents, and the results achieved by the process and apparatus disclosed by the patents.

Specifically, the 497 reactor’s claim 1 of the patent calls for the introduction of a neutralizing agent, aeration, and agitation all positioned generally adjacent to each other. Claim 1 of the 497 patent states:

1. A water treatment apparatus comprising,  
a reaction vessel,  
a source of acidic or metal-bearing water,  
influent pipe means operatively connected to said reaction vessel for delivering said water to be treated from said water source to said reaction vessel,  
aerator means having a shaft extending therefrom into

said reaction vessel said shaft having a discharge end  
        for discharging oxidant,  
    said aerator means having agitation means,  
    said influent pipe means disposed generally adjacent to  
        the discharge end of said aerator shaft,  
    a neutralizing agent feed line means leading from neu-  
        tralizing supply means into said reaction vessel for de-  
        livering neutralizing agent,  
    said neutralization feed line means operatively associat-  
        ed with said aerator shaft such that a discharge end of  
    said neutralizing agent feed line is positioned general-  
        ly adjacent to said agitation means,  
    pump means operatively associated with said reaction  
        vessel,  
    power source means operatively associated with said re-  
        action vessel for energizing said apparatus,  
    effluent discharge pipe means operatively connected to  
    said reaction vessel for discharging the water, and  
    automated metering and control means for delivering  
    said neutralizing agent into said reaction vessel  
        through said neutralizing agent feed line means and ox-  
        idant from said aerator means at substantially the same  
        time, whereby said oxidant impinging upon the water  
        entering said reaction vessel will aerate said water and  
        establish mixing therein so as to enhance efficiency of  
        distribution of said neutralizing agent in said water to  
        reduce reaction time and enhance efficiency of said re-  
        action.

Mr. Dupon acknowledges that the prior art identified in the EPA Manual suggest the opposite of what in fact the 497 and 800 patents disclose. Indeed, the EPA Manual and Mr. Dupon suggest that oxidation not be combined “generally adjacent to” or “simultaneously with” the introduction of a neutralizing agent.

The effectiveness of the 497 reactor is demonstrated by the pilot scale and operational data and a typical example follows:

$$\text{Oxygen Transfer Rate Required} = 7.14 \times 10^{-5} [\text{Fe}^{+2}] Q/P \quad (\text{Eqn, 12 EPA Manual})$$

where,  $[\text{Fe}^{+2}]$  = ferrous iron concentration in the influent (mg/L or ppm)

Q = influent flow rate (gpm)

P = aerator horsepower rating (hp)

Given an example for the actual process described in the 497 Patent (see attached data):

$$[\text{Fe}^{+2}] = 390 \text{ ppm}$$

$$Q = 450 \text{ gpm}$$

$$P = 7.5 \text{ hp}$$

or, then: Oxygen Transfer Rate Required = 1.67 lb/hp-hr (ignoring Mn as a minor component).

This rate of oxygen transfer is well within the rate specified by the manufacturer for design purposes (2.5 to 3.6 lb/hp-hr, see attached specifications - Aeration Industries International, Inc., Chaska, MN), and, using the rate specified by the manufacturer, one would expect to be able to treat a maximum ferrous iron concentration of 840 mg/L for each 7.5 hp aerator used in a properly designed and operated system.

Mr. Dupon recognizes that the aerator rating is the appropriate interpretation of the claim language in the 800 patent providing oxygen concentration rates.

Claim 1 states:

What is claimed is:

1. A method for removing metal compounds selected from iron, manganese, aluminum, zinc, copper, lead, arsenic and chromium from waste water comprising the steps of:
  - (a) adjusting the pH of the waste water to from about 5 to about 12;
  - (b) aerating the waste water;
  - (c) agitating the waste water, where steps (a), (b) and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a metals concentration of from about 50 mg./l. to about 1,000 mg./l.

Again, the results obtained support the reduced residence time provided by the compact and efficient design of the reactor, which sets the 497 patent apart from the prior art, identified.

Accordingly, the 497 reactor causes an instantaneous reaction and oxidation that greatly reduces the residence time required for the precipitation of metals from a waste stream.

Mr. Dupon points out that the EPA MANUAL discloses that "Neutralization, mixing, and aeration take place in the same process unit (figs. 2-3, 7-5)". However, the figures referred to clearly show the recycle of solids back to the neutralization/oxidation reactor (also, see original

Patent #3,738,932). The 497 Patent shows no recycle of solids. It would not be obvious to one skilled in the art that the system described in the 497 Patent would work without the solids recycle in a single reactor. In addition, the patent referred to in the EPA MANUAL (US Patent #3,738,932) shows two reactors, not just one, in the process. More importantly, the method employed by the 497 reactor, namely the intimate contacting of reactants, is not disclosed at all by the EPA Manual. In my opinion therefore, this prior art neither anticipates nor would make obvious the elements claimed the 497 or 800 patents.

Referring to Mr. Dupon's report, Par. 101-104, and commenting in the same order as his claim chart comparing the Patent and the EPA Manual:

1. The chemistry is the same. Looking at the information in Chapter 1 of the EPA Manual in Chap. 1, Fig. 1-1, it appears that the  $Mn^{+2}$  and  $Al^{+3}$  would not precipitate until the pH is above 9 or 10 respectively. Yet, these species are removed at pHs of about 8.5 in the patented reactor.
  - a. See also Claim 18, in which the pH range of 6-9 is identified.
  - b. The patented reactor has more effective oxygen transfer as evidenced by its performance. The aeration technique is not specified on pg 7 of the EPA Manual.
  - c. It is clear that the sludge is recycled (Figs. 2-3 and 7-5). As discussed earlier, this further increases the reactor size, requires recycling precipitated sludge, and moves the reactor out of the "portable" category. Pg. 59 of the EPA Manual deals only with the mixing in individual steps. It is apparent, as Mr. Dupon surmised, that the dissolved oxygen concentration was in fact the oxygen utilization rate.
  - d. These steps are taken out of context of the *entire process*, as the patent deals with the quality flocs generated in this unique reactor system and is dependent on both. It must again be emphasized that this process must be tailored to specific wastes thereby proving a flexible basis for treating wastes of different characteristics.
  - e. See Claims 1d, 3,7,11, 16, 17, and 20. Again, the process is the sum of the parts and the fact that individual steps are practiced; they are not practiced in conjunction with this unique reaction system in these sequences.
2. The EPA Manual (Pg. 135) discusses a filter press. This is a bulky, heavy, labor intensive, batch operation. Use of a filter press would totally defeat many of the objectives of the 497 Patent.
3. This is taken out of context of the overall process. Again, the operation of the individual parts is not the same as the unique and effective operation of this entire reaction – dewatering system.
4. Same as 2.
5. Taken out of context of the entire reactor-dewatering system.
6. Same as 5.
7. Same as 5.

8. Same as 2
9. Same as 2.
10. Same as 2.
11. Same as 2.
12. Same as 3.
13. Same as 2
14. Same as 2.
15. Same as 2.
16. Same as 2.
17. Same as 3.
18. See claim 18 of the 800 Patent. This is consistent with typical EPA discharge requirements and as such represents the best operating range since this would eliminate subsequent pH adjustment.
19. Indeed, the neutralizing agents being used are the state of the art. The chemistry doesn't change. This still doesn't address the unique character of the reactor.
20. It is stated in the Patent (800) that possibly anionic or cationic polymers may be used. Any one skilled in the art recognizes that specific polymers for specific wastes are determined empirically through testing.
21. Same as 20.
22. Same as 20.
23. This concentration range is determined by experience and testing.
24. Same as 3.
25. Same as 3.

In reference to Mr. Dupon's paragraphs 105 and 106, the individuals units, a reactor, and aerator, or clarifier, can be built based on the EPA Manual. Yet the synthesis of this overall process with its unique reactor of demonstrated performance could not have been established solely based on the EPA Manual. U.S. Patent 4,834,878 is not the same as the 800 Patent. The 800 Patent delineates dewatering techniques covering a wide range of conditions suitable for the specific wastes considered. Remarkably, this process operates continuously with the production of quality sludge that can be disposed directly. The equipment is compact and can handle large volumes of water, and remove metals from the water quickly and effectively. This method did not exist before the patent application was filed.

Referring to Mr. Dupon's paragraph 107, and commenting in the same order as the claim chart comparing the Patent and the EPA Manual:

3.
  - a. Mr. Dupon's statement that "AMD is a dilute solution of sulfuric acid" is incomplete and most misleading. The statement needs to be completed (as stated in the EPA Manual) "...and iron sulfate with iron in the ferrous and/or ferric form." Even this is incomplete but not misleading.
  - b. Fig 7-5 in the EPA Manual is quite different with sludge recycling.
  - c. A submerged turbine aerator is different than the aerator used in the 497 and 800 Patents.

- d. Same as c.
- e. It is apparent that any AMD process would have associated piping and pumps for the transfer of liquids and appropriate solids handling equipment.
- f. Same as e.
- g. Same as e.
- h. Same as e.
- i. Same as e.
- j. The aerator used in the Patent is different. The EPA Manual indicates a surface aerator.
- k. The agitator is different than any of the four types discussed in the EPA Manual. Again, the overall design concept and method are quite different.
- l. The integrated design utilizes a number of individual type units.
- m. Same as e.
- n. Same as j.

- 4. Same as 3k.
- 5. Same as 3b.
- 6. Same as 3a.
- 7. Again, the synthesis of this process described in the Patent is unique. In my opinion one skilled in the art would not think to combine the individual parts, in this uniquely configured method and system. This system achieves the goals of the Patent.
- 8. Same as 7.
- 9. The EPA Manual deals only with sodium hydroxide as a chemical, its properties, and does not address its use in the specific manner in which it is used in the patented processes (497 and 800).

Regarding Par. 108, the Patent description speaks for itself.

Regarding Pars. 109 and 110, the combination of the small tank, its low residence time of wastewater, and effective removal under controlled conditions, were not obvious at the time of the invention. This is evidenced by the successful achievement of the goals as set forth in Patent 497 and 800. Accordingly, I conclude that the patents 497 and 800 represent the unique synthesis, design, and execution of a process that more effectively treats AMD and similar type wastewater than the prior art.

#### Anticipation and Obviousness - Other references

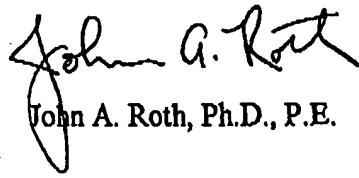
Having reviewed the other prior art referenced in Mr. Dupon's report, attached as Attachment B to his report, none of that prior art individually anticipates, that is identifies the synthesis of all the elements identified in the 497 or 800 patents alone. Nor does any of the prior art render the claimed inventions methods and apparatus obvious, that is a person of ordinary skill in the art after reviewing such prior art would conclude that the invention was obvious. The

primary reason for the opinion is that the inventions claimed in the 497 and 800 patents obtain a significantly reduced retention time for the neutralized waste stream and the heightened dewatering ability that is made possible by such instantaneous reaction. Additionally, the prior art does not anticipate or make obvious the compact and portable aspects of the invention, nor does the prior art anticipate or make obvious the cost effective treatment method disclosed by the patents.

Conclusion

Having reviewed all the material supplied with Mr. Dupon's report, as well as his report, operational data, the patents, and prosecution histories of both patents, I conclude that one skilled in the art would be able to construct and operate the inventions set forth in the patents to obtain their stated goals. I further conclude that none of the information supplied, alone or combined, would suggest to one skilled in the art that the elements combined in either patent would produce the reduced residence times and quick and effective removal of metals and dewatering of sludges in the compact apparatus described. Prior to April 1987, nothing in the industry, as disclosed by the literature, achieved the reduced residence times and compact design achieved by the 497 reactor design and method. Prior to December 1994, nothing in the industry, as disclosed by the literature, produced the reduced residence times, and quick and effective removal of metals and dewatering of sludges as achieved by the 800 patent's designs and methods.

Sincerely,



John A. Roth

John A. Roth, Ph.D., P.E.



U.S. Department of Justice

CRE  
154-97-21

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July 7, 2000

**VIA FEDERAL EXPRESS**

C. William Kenny, Esq.  
The Tarasi Law Firm, P.C.  
510 Third Avenue  
Pittsburgh, PA 15219-2191

Re: *Chemical Separation Technology, Inc., et al. v.*  
United States, Court of Federal Claims No 97-21C

Dear Mr. Kenny:

I have enclosed our supplemental expert disclosure, as well as prior art, all Bates numbered D026313-331.

Very truly yours,

Cameron Elliot  
Attorney  
Commercial Litigation Branch  
Civil Division

Enclosures

IN THE UNITED STATES COURT OF FEDERAL CLAIMS

CHEMICAL SEPARATION TECHNOLOGY, )  
INC., and SANFORD M. STEVENSON, )  
Plaintiffs, )  
v. ) No. 97-21C  
THE UNITED STATES, ) Judge Francis M. Allegra  
Defendant. )

SUPPLEMENTAL DECLARATION OF DANIEL DUPON

Daniel Dupon, of Fort Collins, Colorado, declares under penalty of perjury that the following is true and correct:

1. With regard to mine wastewater chemistry, although I agree that the hydrolysis of dissolved metals in solution is an essential element to consider in treating acid mine drainage, as I mention in paragraph 49 of my Declaration, my discussion in paragraph 7 was intended only to show the relevance of pH to the acidity or alkalinity of a solution. The statements were not intended to describe how to calculate or measure the quantity of acidity in a solution, but to indicate the correlation of low pH values to acidic solutions and high pH values to alkaline solutions. Discharge pH is an important parameter in itself because high  $H^+$  and  $OH^-$  concentrations (corresponding to low and high pH, respectively) have both direct and indirect toxic affects and are therefore regulated independently of acidity. The equation on page 2 of the plaintiffs' report is a

modification of the acidity equation applicable to carbonate solutions found on page 184 of Water Chemistry, by Snoeyink and Jenkins. The additional metal term represents acidity generated from hydrolysis of dissolved metals in solution. The equation in the plaintiffs' report should include brackets around the metals term, as with the first four terms on the right side of the equation, to indicate molar concentration; thus: Acidity =  $2[H_2CO_3] + [HCO_3^-] + [H^+] - [OH^-] + \Sigma n [Me^{+n}]$ . When calculating acidity for a given AMD solution, the most accurate method is to use the valence state (represented by "n") of the metal that will precipitate (i.e.,  $Fe^{3+}$ ), not the state of the untreated metal in solution (i.e.,  $Fe^{2+}$ ), particularly in a treatment scheme that utilizes metal oxidation. The symbol " $\Sigma$ " indicates that the last term is actually the sum of the individual metal concentrations multiplied by their post-precipitation valence states.

2. I disagree that the term "metal compounds" in the '800 patent should be read to refer only to metal oxyhydroxides. Lead carbonate, for example, can be precipitated when a carbonate-based reagent (such as calcium carbonate) is used as the neutralizing chemical, as described in U.S. Patent No. 4,652,381 ("Inglis"), and forms a less hydrous solid than lead hydroxide (i.e., a higher quality floc). Also, the solubilities of metal carbonates are sometimes lower than the solubilities of metal hydroxides, depending on the process pH, which dictates their

solubility and subsequent precipitation. Although the Example in the '800 patent alludes to the generation of a metal hydroxide sludge, not all metals listed in claim 1 are removable as oxides or hydroxides without invoking treatment techniques beyond the steps described, as I explain in paragraph 23 of my Declaration.

3. I agree that trivalent chromium precipitates upon neutralization, that it can be chemically reduced in the presence of ferrous iron, and that it oxidizes relatively slowly in the presence of atmospheric oxygen. Thomas J. Sorg, "Treatment Technology to Meet the Interim Primary Drinking Water Regulations for Organics: Part 4," Water Technology/Quality Journal AWWA 454, 455 (August 1979) (D026275). Hexavalent chromium is often of greater concern for water quality because trivalent chromium is so insoluble it is normally found at dissolved concentrations close to EPA drinking water limits. Since claim 1 of the '800 patent recites chromium as a removable metal, without distinguishing between trivalent and hexavalent forms, it should be capable of removing either form even if present alone in solution. One of the best demonstrated removal methods for hexavalent chromium is chemical reduction to trivalent chromium, followed by coprecipitation or precipitation of the trivalent chromium. Francoise C. Richard and Alain C. M. Bourg, "Aqueous Geochemistry of Chromium: A Review," 25 Water Research 807, 810 (1991) (D026267); J. M. Philipot, F. Chaffange, and J. Sibony,

"Hexavalent Chromium Removal from Drinking Water," 17 Water Science Technology 1121, 1124-1129 (1984) (D026290-95); Sorg, p. 460 (D026280). Such a procedure is not described or even contemplated by the '800 patent, and my conclusion that the '800 patent is not enabled and fails the written description and best mode requirements is based in part on this omission.

4. Although copper has two oxidation states, and is therefore capable of redox behavior, oxidation is generally not required for its removal because copper in the lower oxidation state is unlikely to be found in AMD solutions where the pH is less than 5. As a result, one skilled in the art of water treatment would recognize that merely adjusting wastewater pH is normally all that is necessary to precipitate copper, even in the absence of coagulants/coprecipitants such as iron. An example of such a procedure is described in the Inglis patent, col. 7, 11. 47-56, where copper, zinc, and lead are precipitated using carbonate without aeration at a pH between 8.0 and 9.0.

5. I disagree that the term "about" in reference to a pH of "about 5 to about 12" is sufficiently precise to discern the scope of claim 1 of the '800 patent. First, although the selection of a lower pH limit of about 5 could be based on oxidation and precipitation of iron, or simply precipitation of aluminum, nothing in the patent says that this is the basis. Second, if the advantage of the claimed invention over the state

of the art is its rapid reaction rate, a pH limit of about 5 is not optimal, since ferrous iron oxidizes and precipitates more rapidly at pH 6 and higher. Furthermore, as the plaintiffs point out on page 4 of their report, a difference of 1 pH unit represents an "exponential" (10-fold) difference in concentration, and a change of 0.3 units represents a two-fold difference, both of which can have significant implications when optimizing iron oxidation rates, particularly in water treatment processes that have limited detention times. Therefore, because the basis for a pH range of "about 5 to about 12" is unknown, my opinion remains that one skilled in the art would not be able to tell whether a particular pH is "about 5" or "about 12."

6. I agree that the expression "provide a dissolved oxygen concentration at from about 0.001 lb./hr. to about 70 lbs./hr" could also be interpreted as "oxygen utilization rate," assuming that "oxygen utilization rate" means the actual addition rate of dissolved oxygen that is required in the oxidation reaction, since one skilled in the art would know that the aerator should be operated so as to match the "oxygen utilization rate" to the aerator output. Plaintiffs' Report, p. 9. However, such an interpretation makes claim 1 of the '800 patent even more indefinite, because, for example, assuming the maximum listed flow rate and metals concentration (for manganese alone in solution), the "oxygen utilization rate" may exceed 70 pounds per

hour. Using the same assumptions applied for other chemically-reduced metals, and further assuming that dissolved oxygen efficiently oxidizes trivalent chromium, the "oxygen utilization rate" would exceed 100 pounds per hour. The plaintiffs also state that "the aerator rating is the appropriate interpretation." Aerator rating refers to the aerator's capacity to impart oxygen as a function of input power for a specific test vessel configuration, often measured in pounds of oxygen per horsepower per hour, a parameter that is not necessarily the same as the "oxygen utilization rate", the oxygen addition rate, or the dissolved oxygen concentration. I cannot clearly determine the relevance of "Equivalent Oxygen Concentration," as defined in the plaintiffs' report. It has little chemical or engineering significance, because it does not take into account the aerator transfer efficiency and because the oxygen concentration of 99.8 mg/L calculated in the example cannot be achieved under typical operating conditions. This is exactly why the aerator configuration shown in the preferred embodiment of the '497 patent is unlikely to be effective for oxidizing metals that are present at high concentrations, since the described aerator configuration will contact the influent water for such a short period that the claimed invention theoretically must rely on achieving an unrealistically high dissolved oxygen concentration at the point of application.

7. I disagree that the "automated metering and control means" in claim 1 of the '497 patent is limited to an agitator, a pH probe, and a metering pump. The agitator shown is not controlled by the metering and control means. More to the point, no structure is shown for delivering "oxidant from said aerator means at substantially the same time" as the neutralizing agent. I list some mechanisms for regulating oxidant delivery in my Declaration, all of which operate in different ways but none of which are shown in the patent.

8. "Introducing a source of acidic or metal bearing water into a treatment unit" may be achieved by gravity flow or pumping, and both techniques feed raw wastewater into the reactor. However, pumping requires external power for the pump and provides a driving pressure for conveyance of water while gravity flow relies only on gravitational forces and may be employed in an open channel. In my opinion, these two techniques do not perform the function of introducing water in substantially the same way.

9. I disagree that the EPA Manual suggests that oxidation not be combined generally adjacent to, simultaneously with, or in the same area of the treatment unit as, neutralization agent addition. Page 48 of the EPA Manual discusses some factors to consider in selecting the oxidation method, including operating pH, detention time, and ferrous iron concentration, and even

suggests that in some cases oxidation should precede neutralization. Figures 2-2 and 7-5 of the EPA Manual show alternate configurations, one with aeration and neutralization occurring in the same treatment unit and one without. In addition, figure 7-5 also shows the recycling of sludge, which has two purposes. First, it promotes creation of denser precipitates, which makes disposal easier. Second, it allows for more cost-effective use of neutralizing agent, since the sludge may have alkaline properties. Both the EPA Manual and U.S. Patent No. 3,738,932 show mixing of recycled sludge with lime in a chemical preparation reactor, followed by addition of the mixture to the AMD-aeration-neutralization reactor. The mixture (instead of the lime alone) thus acts as the neutralizing agent, allowing for a lower rate of lime consumption that depends on the mode of operation.

10. I agree that acid mine drainage contains solutes other than sulfuric acid, including dissolved metals which increase acidity, but the presence of dissolved sulfuric acid alone is sufficient to categorize AMD as "acidic" as the term appears to be used in the '497 patent. As I discuss above and in paragraph 49 of my Declaration, however, the term "acidic" is used ambiguously in the patent; broadly, it could mean "having a low pH," "containing dissolved metals that produce acidity upon hydrolysis," or both at the same time.

11. Whether a submerged turbine aerator is different from the aerator used in the '497 patent depends on how the patent's claims are understood. The EPA Manual (p. 77) describes a submerged turbine aerator as having an air sparger (or diffuser) near the bottom of the treatment unit and one or more turbine impellers above it. Since a turbine impeller normally is powered by a shaft, as shown in figure 5-2 (p. 76), a submerged turbine aerator has a diffuser, a shaft, and an agitator/impeller, just as the '497 patent's aerator does. It is true that a submerged turbine aerator may not have the air diffuser mounted on the shaft; a typical submerged turbine aerator is shown in unit 16 of U.S. Patent No. 5,013,453, part of the PTO file for the '800 patent. Nonetheless, if the '497 patent's aerator means is not interpreted to be limited to the specific aerator shown in figure 3, it is my opinion that the aerator claimed in claims 1 and 3 is described in the EPA Manual.

12. Except for claims 1, 2, and 9 of the '497 patent, no claim of either patent references reduced detention time, an instantaneous reaction, or cost effectiveness of treatment, and claims 1 and 2 only speak ambiguously of "efficiency." Nor does any claim require that the inventions be compact and portable. I therefore conclude that these features are irrelevant to the claimed inventions.

13. It is still my opinion that other prior art references either anticipate the claims of the '800 and '497 patents or would have made them obvious, although (as with the EPA Manual) this depends on how the claim language is understood. As shown in the claim chart below, whether the Inglis patent anticipates any claims of the '800 patent depends on the interpretation of the claim language:

A method for removing metal compounds selected from iron, manganese, aluminum, zinc, copper, lead, arsenic and chromium from waste water comprising the steps of:	Iron is removed (col. 3, ll. 52-53). Lead, copper, and zinc are removed (col. 3, l. 47).
(a) adjusting the pH of the waste water to from about 5 to about 12;	"pH of the waste water contained in the treatment tank 28 [is] maintained at a pH of from about 4.8 to 5." Col. 6, ll. 39-41.
(b) aerating the waste water;	"Air is sparged continuously into the interior of treatment tank 28" Col. 6, ll. 11-12.

<p>(c) agitating the waste water, where steps (a), (b), and (c) are carried out simultaneously in a reaction tank and waste water is aerated in said reaction tank to provide a dissolved oxygen concentration at from about 0.01 lb./hr. to about 70 lbs./hr. at a waste water input flow rate of from about 50 gal./min. to about 500 gal./min. for a metals concentration of from about 50 mg/l. to about 1,000 mg/l.</p>	<p>"Air is sparged continuously into the interior of treatment tank 28 through line 38 to effect some mixing of the liquid" Col. 6, ll. 11-13.</p> <p>"pH of the waste water contained in the treatment tank 28 [is] maintained at a pH of from about 4.8 to 5." Col. 6, ll. 39-41.</p> <p>"the present method treats 1,200 gallons a day" Col. 3, ll. 64-65.</p> <p>"Total lead in the waste water can run as high as 500 ppm." Col. 5, ll. 40-41.</p> <p>"Iron is normally present at levels about 50 ppm while copper can be as high as 10 ppm." Col. 5, ll. 43-44.</p>
<p>(d) then adding a flocculating agent polymer selected from a group consisting of cationic and anionic polymers to the water and allowing floccules including said metal compounds to form; and</p>	<p>"an anionic polyelectrolyte flocculant contained in tank 92 is pumped via line 94 into the final treatment tank 50." Col. 7, ll. 58-60.</p>
<p>(e) then separating said floccules including said metal compounds from the water.</p>	<p>"the contents of final treatment tank 50 are allowed to settle" Col. 7, ll. 64-65.</p>

14. The dissolved oxygen concentration is not given, but one skilled in the art would know how to determine the dissolved oxygen addition rate (assuming that is to what "dissolved oxygen

concentration" refers in claim 1 of the '800 patent) needed to oxidize the iron in Inglis. This rate falls below the lower limit of "about 0.01" pounds per hour, as do the initial pH and the flow rate. Alternatively, one skilled in the art would know that the required dissolved oxygen would have to be about 7 mg/L to achieve complete oxidation of 50 mg/L of iron. One skilled in the art of water treatment would also recognize that the purpose of adding anionic polyelectrolyte flocculant is to allow flocculated particles of precipitated lead, copper, and zinc to form. In short, assuming the pH, dissolved oxygen concentration/aeration rate, and flow rate of claim 1 of the '800 patent extend below their lower limits, Inglis anticipates claim 1.

15. Assuming these elements do not extend below their lower limits, I believe one skilled in the art of water treatment would have known on May 25, 1993, by reading Inglis, that iron may be oxidized at a pH of 5, that the Inglis system could be operated at a higher flow rate of 50-500 gallons per minute, and how to calculate the theoretical oxygen demand and corresponding concentration for oxidization of 50 mg/L of iron at such a flow rate. This teaching is contained in the EPA Manual, pp. 3, 74. I therefore conclude that claim 1 of the '800 patent would have been obvious to one of ordinary skill in the art of water

treatment, on the assumption that the rate and concentration ranges do not extend below their lower limits.

16. Similarly, I conclude that the following claims would also have been obvious if they are interpreted in the manner indicated above:

2. claim 1, plus step (f) (further dewatering the floccules separated in step (e))	belt filter 64
6. claim 2, where step (e) separation is conducted by means of a clarifier	treatment tank 50
15. claim 2, where step (e) separation is conducted by means of a settling pond	treatment tank 50
21. claim 1, wherein the polymer is an anionic polymer which is used for primary clarification purposes	anionic polyelectrolyte flocculant
22. claim 1, wherein the polymer is an anionic polymer which is used for settling purposes	anionic polyelectrolyte flocculant

17. Continuous belt filter 64 is sufficiently like a conventional belt filter press (as shown in, e.g., Nalco Water Handbook, pp. 9.46-9.49) that one skilled in the art would have known to use a belt filter press in lieu of a belt filter. Moreover, tank 50 is referred to in Inglis as a "final treatment tank," which one skilled in the art would know from the EPA

Manual is equivalent to both a settling pond and a clarifier, depending on site conditions and cost considerations. EPA Manual, c. 6. Other claims would also have been obvious in light of, for example, U.S. Patent No. 4,834,878 ("Anderson"), which describes a rotary thickener in series with a belt filter press, with polyelectrolyte addition, for the purpose of sludge dewatering.

18. As with the '800 patent, whether Inglis anticipates the claims of the '497 patent depends on interpretation of the claim language:

3. A method of treating acidic water comprising,	"the water [has] a pH below 2" Col. 5, ll. 42-43.
providing a treatment unit comprising aerator means,	tank 28 and aerator 38
said aerator means having a shaft extending therefrom into said treatment unit	aerator 38 with shaft extending into tank 28
and said aerator means having agitation means,	"Air is sparged . . . to effect some mixing of the liquid" Col. 6, ll. 11-13
influent pipe means disposed generally adjacent to the discharge end of said aerator shaft	pipe 14 in the same tank 28 as aerator 38
and neutralizing agent feed line means disposed generally adjacent to the exit of said aerator means,	pipes 20 and 24 in the same tank 28 as aerator 38

providing means for introducing a source of acidic or metal-bearing water into a treatment unit,	"the water [has] a pH below 2" Col. 5, ll. 42-43. pipes 12 and 14, pump 16
introducing said water to be treated into said treatment unit,	"Waste water collected in sump 10 is pumped to vessel 28 via line 12" Col. 5, ll. 55-56.
introducing a neutralizing agent into said treatment unit,	"Calcium carbonate . . . is pumped via line 20 into the interior of treatment tank 28" Col. 6, ll. 19-21.
introducing oxidant at generally the same point at which said neutralizing agent is introduced into the water to be treated,	"Air is sparged . . . to also provide an adequate supply of oxidizing agent (in this case O <sub>2</sub> )" Col. 6, ll. 11-14
agitating said water, oxidant, and neutralizing agent,	"the major mixing of liquid in treatment tank 28 is provided by recirculation of liquid through line 32 by pump 36." Col. 6, ll. 16-18.
providing a means for receiving precipitants from said mixture, and	"Heavy metal carbonates . . . collected in the bottom of final treatment tank 50" Col. 7, ll. 67-68. tank 28
discharging treated water from said treatment unit.	discharge pipes 44, 98, 108
4. Claim 3, wherein said primary oxidant source is air.	"Air is sparged . . . to also provide an adequate supply of oxidizing agent (in this case O <sub>2</sub> )" Col. 6, ll. 11-14
5. Claim 3, wherein the neutralizing agent and the oxidant are substantially simultaneously introduced into said water.	aerator 38 and feed line 14 terminate in tank 28

19. Assuming that the "agitation means" is not limited to a propeller, that the influent pipe means has no particular structure inside the treatment tank, that the "means for introducing" is a pump, that "generally the same point" means "in the same area of the tank," that either tank 28 or final treatment tank 50 count as means for receiving precipitants from said mixture, and that "substantially simultaneously" means "in the same area of the tank," Inglis anticipates claims 3, 4, and 5 of the '497 patent. If the agitation means is limited to a propeller, then the aerator shown in U.S. Patent No. 4,240,990 ("Inhofer") meets the requirements of both the aerator means and the agitation means. Since Inhofer suggests using this aerator in "water treatment facilities" (col. 1, ll. 10-11), and because the Inglis aerator is very similar (they both possess a shaft and produce agitation near their discharge), one skilled in the art would know to replace the Inglis aerator with the Inhofer aerator. So interpreted, therefore, I conclude that claims 3, 4, and 5 of the '497 patent either are anticipated or would have been obvious in light of Inglis and Inhofer. I also conclude that, except for the "power source means" and "automated metering and control means," all the elements of claim 1 are described in Inglis, given this claim interpretation. Since it would have been obvious even to one not skilled in the art to incorporate a power source into Inglis, and on the assumption that the

"automated metering and control means" is simply a pH probe connected to a metering pump (which are also found in Inglis), I conclude that claim 1 would have been obvious, also.

I declare under penalty of perjury that this supplemental declaration is true and correct.

Dated: June 30, 2000



DANIEL DUPON

CERTIFICATE OF SERVICE

Pursuant to 37 C.F.R. § 1.248 I declare that a true copy of the foregoing "PUBLIC PROTEST PURSUANT TO 37 C.F.R. § 1.291" was served by first-class mail, postage prepaid, this 31st day of July 2001 to:

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

RE Application of )  
SANFORD M. STEVENSON )  
Serial No. 09/652,272 )  
Filed: August 31, 2000 )  
FOR: METHOD FOR REMOVING METAL )  
COMPOUNDS FROM WASTE WATER )

Commissioner for Patents  
Washington, D.C. 20231

Art Unit 1724

Examiner: P. Hrusci

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TC 1700

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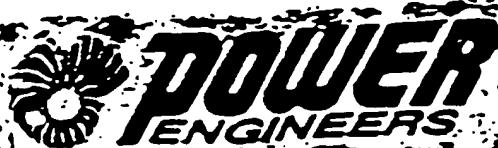
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July 31, 2001

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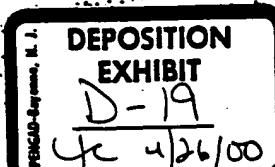
CHEMICAL SEPARATION TECHNOLOGY, INC.  
VANCOUVER, WASHINGTON

REVIEW AND DOCUMENTATION  
OF THE CST INDUSTRIAL  
WASTEWATER TREATMENT  
SYSTEM

FEBRUARY 27, 1991

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EXHIBIT

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0005620

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## APPENDIX

- A: Cost Breakdown for 500 GPM Case
- B: Cost Breakdowns for All Four Cases
- C: Drawings

## REVIEW AND DOCUMENTATION OF THE CST INDUSTRIAL WASTEWATER TREATMENT SYSTEM

The information and opinions expressed in this report are based on information obtained by the writer from:

- (1) field visits to the Keystone, Conemaugh, and LTV coal refuse sites
- (2) from discussions with Mr. Sandy Stevenson (CST, Inc.) and Mr. John Anderson (Thickener Technology, Inc.)
- (3) information contained in the technical literature.

### 1. INTRODUCTION

The CST system offers an alternative to lime-based, industrial wastewater treatment technology, and has application for treating leachates derived from

- (1) coal refuse piles
- (2) coal stockpiles
- (3) working and abandoned coal and base-metal mines
- (4) acid-generating mine waste dumps
- (5) other industrial sources which require neutralization, oxidation and precipitation

The CST system may also have applications where a gas (or gases) and a liquid reactant act on a component of the solution. An example might be detoxification of cyanide-containing solutions.

Currently, the CST system is used to treat leachates from the LTV and Keystone coal refuse landfill sites in Western Pennsylvania.

The CST system achieves rapid acid neutralization and metal precipitation of coal refuse leachates by reacting the leachate with sodium hydroxide and air-derived oxygen in a patented reactor at atmospheric pressure.

A commercial aerator, which is mounted on the reactor, draws air continuously into the reaction zone in the form of very fine bubbles. The oxygen oxidizes ferrous iron into ferric iron which precipitates at a lower pH and in a form less voluminous than ferrous iron. Although the aeration rate is fixed, the reactor can accommodate up to two aerators.

Oxidants other than oxygen, such as hydrogen peroxide, sodium hypochlorite, ozone, permanganate, etc., may be used.

Concentrated sodium hydroxide (solutions up to 50 percent NaOH may be used) is used as the neutralizing base. This strong base completely dissociates and provides 100 percent alkalinity for acid neutralization (i.e., all of its hydroxyl ions are available for neutralization).

Unlike lime-based systems, it is not necessary to recycle "sludge" to the CST reactor to increase alkali utilization or to promote sludge thickening. Also, unlike lime-based systems, gypsum is not precipitated from solution.

A programmable logic controller (PLC) is used to precisely meter sodium hydroxide to the reactor based on the leachate flowrate and the desired neutralization pH. This important feature minimizes sodium hydroxide consumption and allows nearly complete metal precipitation.

The reactor effluent contains insoluble metal hydroxide and salts, collectively termed precipitants. Typically, the effluent contains from 0.5 to 1.5 percent solids by weight, depending upon the amount of metals in the leachate.

Although the acidity and level of dissolved metals in the reactor effluent comply with regulatory discharge requirements, discharge of the effluent to a watershed is not permitted without first removing the precipitants and suspended solids.

The only exception to this is if the effluent can be discharged to abandoned underground mine workings (such as at the LTV site).

Therefore, the CST system will usually include additional steps or operations which treat the reactor effluent for solids recovery and disposal. This is also true of lime-

based systems. Such steps increase the capital and operating cost of the treatment systems.

Operations which remove and concentrate the precipitants include various forms of gravity sedimentation and filtration devices.

For example, CST utilizes a rotary sludge thickener and a belt filter press at the Keystone leachate treatment plant to increase the solids concentration from about 1 to 25 percent solids by weight.

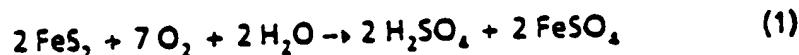
Metal hydroxides are difficult to dewater by mechanical methods due to their extremely small particle size, large surface-to-weight ratio, and their natural affinity for holding water.

To effect solids concentration and recovery, flocculants are used to bind and hold the particles together while their mass undergoes dewatering.

The final step in this process is disposal of the dewatered precipitants. This is done at the Keystone and Conemaugh plants by loading the dewatered material into trucks for disposal at the refuse landfill.

## 2. PROCESS CHEMISTRY

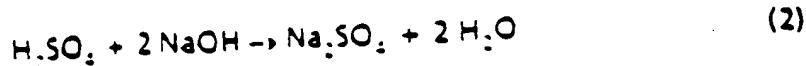
Acidic leachates are produced when pyrite-containing coal seams, coal refuse from preparation plants, or base-metal ores or wastes react with oxygen and water to produce sulfuric acid and ferrous sulfate. The exact reaction scheme depends upon pH and whether thiobacillus ferrooxidan bacteria are present. In general, the production of acid and ferrous iron is given by equation 1:



The methods for treating the products of this reaction are neutralization, oxidation, and precipitation.

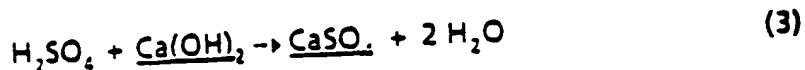
## 2.1 Neutralization

Sodium hydroxide neutralization proceeds according to equation 2:



On a weight basis, one pound of sulfuric acid reacts with 0.82 pounds of sodium hydroxide.

When hydrated lime is used, the reaction is given by equation 3:



Although only 1 mol of hydrated lime is required to neutralize 1 mol of acid, almost the same weight, 0.76 lbs, is required to neutralize 1 lb of acid. This is because the molecular weight of hydrated lime is about twice that of sodium hydroxide.

Sodium hydroxide neutralization (equation 2) is stoichiometric, which is not the case when lime is used. Calcium hydroxide is quite insoluble and reacts principally as a solid with the acid. Neutralization is therefore slower when lime is used.

Another factor affecting lime-based systems is fouling of the lime with reaction products, notably gypsum and iron precipitates. These materials coat the lime particle surfaces, hindering reaction with the acid.

It is generally accepted that from 1.5 to 2 times more calcium hydroxide is required to supply the hydroxyl ions required by reaction 3. Therefore, the neutralization of 1 pound of acid can require from 1.1 to 1.5 pounds of hydrated lime.

Burnt lime (CaO) is used to make calcium hydroxide by slaking. It currently costs about \$80/ton delivered. This compares to \$340/ton for caustic in the Pittsburgh area (or \$450/ton in southwestern Montana).

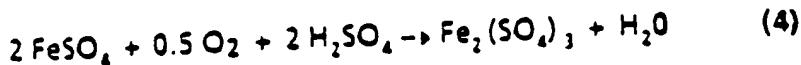
Therefore, the cost of neutralizing 1 pound of acid is \$0.14 for caustic and from \$0.04 to \$0.06 for lime. This comparison does not include the small additional cost of slaking burnt lime to produce hydrated lime.

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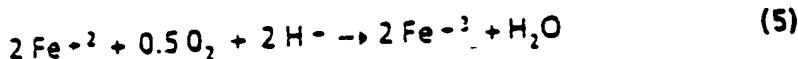
It is fair to say that sodium hydroxide neutralization costs 2.5 to 3.5 times more than lime neutralization, based only on alkali costs.

## 2.2 Oxidation

Iron oxidation is accomplished by providing oxygen according to equation 4;



or according to equation 5, if sulfate ion is omitted;



These equations show that 1 pound of oxygen oxidizes nearly 7 pounds of ferrous iron.

Laboratory tests have shown that the rate of oxidation is directly proportional to the concentration of ferrous iron and dissolved oxygen and inversely proportional to the square of the hydrogen ion concentration; i.e., as:

$$\frac{d[\text{Fe}]}{dt} = k \frac{[\text{Fe}][\text{O}_2]}{[\text{H}]^2} \quad (6)$$

This relationship shows that the rate of iron oxidation is especially sensitive to the solution pH. Generally, rapid iron oxidation occurs above pH 7.5.

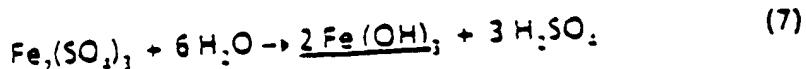
The oxidation rate also depends upon the availability of dissolved oxygen in solution. Oxygen solubility varies with temperature, pressure, and the amount of dissolved matter. Typically, it ranges from 6 to 8 mg/L.

If the level of dissolved oxygen decreases, or if oxygen cannot be supplied at the rate at which it is being consumed, the oxidation rate decreases.

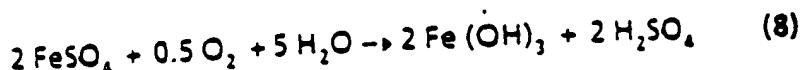
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### 2.3 Precipitation

Iron precipitation occurs when ferric iron reacts with water. This is referred to as hydrolysis:



Since oxidation and precipitation occur essentially together in the CST reactor, the overall reaction can be given by combining equations 4 and 7:



This equation shows that one pound of ferrous iron consumes 0.14 pounds of oxygen. If oxidation is complete, 1.9 pounds of ferric hydroxide and 1.7 pounds of sulfuric acid are formed.

Equations 2 and 8 provide a means for estimating sodium hydroxide and oxygen, given the leachate pH and iron content.

In addition to chemistry, information on reaction kinetics such as the rate of iron, manganese, and aluminum removal and oxygen consumption in terms of pH and leachate flowrate (i.e., residence time) would be desirable. At present, this information is lacking.

### 3. CALCULATED QUANTITIES

An important parameter in selling CST's technology is the ability to predict the cost of reagents, especially sodium hydroxide, for a particular application.

Obviously, the best way to obtain this information is to run the leachate through a small CST reactor and to carefully monitor reagent usage and aeration power, perform influent and effluent analyses, etc.

In lieu of this, the following section presents some ways for calculating sodium hydroxide usage based on the leachate analyses and the reaction chemistry presented in Section 2.

P005627

To illustrate the method, data collected during the field visit to the Keystone site on January 28, 1991, shown in Table 1, are used.

During the sampling period, the CST reactor was fed from two leachate sources: one was directly from the refuse pile runoff, referred to as "weir" in Table 1, and the otherwise from the leachate holding pond. These flow rates were used to weigh the average leachate feed parameters.

Not shown in Table 1 is the pH of the reactor effluent, which was 8.8 during the sampling period.

### 3.1 NaOH Consumption

Sodium hydroxide is consumed in two ways: (1) to neutralize the leachate acidity and (2) to neutralize the acidity generated during the oxidation of iron (equation 8).

P005628

Table 1. Keystone CST Plant Data  
for January 28, 1991

<u>Parameters</u>	Leachate			
	<u>From Weir</u>	<u>From Pond</u>	<u>Plant Total</u>	<u>Plant Effluent</u>
Flow, gpm	90	140	230	230
Air, lb O <sub>2</sub> /HP-hr			3	
Aerator HP			7.5	
NaOH (20%), gal/wk			13,500	
Floc #1, gpm			4	
Floc #1, lbs/gal			0.019	
Floc #2, gpm			2	
Floc #2, lbs/gal			0.01	
Op. time, hrs/day			20	
 pH	2.9	2.9	2.9	7.1
Iron, mg/L	1,200	2,000	1,687	1
Manganese, mg/L	10	18	17	1
Aluminum, mg/L	136	100	114	17
Sulfate, mg/L	2,328	6,800	5,050	2,350
Acidity, mg CO <sub>3</sub> /L	4,100	4,000	4,040	0
Alkalinity, mg CO <sub>3</sub> /L	0	0	0	95
Sus. solids, mg/L	7	3,068	1,870	12

The leachate acidity, in mol/L, is calculated from equation 9:

$$[\text{H}^+] = 10^{-\text{pH}} = 10^{-2.9} = 0.0013 \text{ mol H}^+/\text{L} \quad (9)$$

This concentration of acidity could be converted to sulfuric acid, which could then be converted to NaOH by equation 2.

A simpler way is to recognize that 1 mol of H reacts with 1 mol OH to form 1 mol of water. Therefore, 0.0013 mol H is equal to 0.0013 mol OH which is also equal to 0.0013 mol NaOH. Converting to lbs NaOH/gal gives:

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$$(0.0013)(40)(3.785)/453.6 = 0.0004 \text{ lb NaOH/gal}$$

from which the daily NaOH consumption is calculated as:

$$(0.0004)(230)(60)(20) = 116 \text{ lbs/day.}$$

Equation 8 is used to determine the acidity generated by iron oxidation. It is assumed that the iron content of the leachate is all ferrous iron and that oxidation to ferric acid is complete.

First, the iron in the leachate, 1,687 mg Fe/L, is converted to mols/L

$$\frac{1,687}{(230)(60)(20)} = 0.0302 \text{ mols Fe/L}$$

Equation 9 is used to convert this iron to equivalent sulfuric acid, noting that 1 mol of iron equals 1 mol of  $\text{H}_2\text{SO}_4$ . The result is expressed as lbs acid/gal:

$$(0.0302)(98)(3.785)/453.6 = 0.0247 \text{ lb H}_2\text{SO}_4/\text{gal}$$

Converting to NaOH usage gives:

$$[(0.0247)/98](2)(40)(230)(60)(20) = 5,565 \text{ lbs/day}$$

Adding this to the previously calculated caustic, 116 lbs/day, gives a total caustic demand of 5,681 lbs/day.

These calculations show that 98% of the caustic is consumed in neutralizing the acid generated by the oxidation reaction.

The weight of sodium hydroxide is converted to gallons of 20% caustic by dividing the pounds of caustic by 2.045 lbs/gal:

$$5,681/2.045 = 2,778 \text{ gals/day}$$

If the plant operates 5 days per week, the probable weekly caustic consumption is:

$$2,778 \div 5 = 13,890 \text{ gals 20\% NaOH per week}$$

This calculated figure agrees well with the weekly usage reported in Table 1 based on historical data provided by Mr. Don Knight, operator at the Keystone plant.

The unit caustic usage is found by dividing the demand by the leachate flow:

$$\frac{5,681}{(230)(60)(20)} = 0.0206 \text{ lb NaOH/gal leachate}$$

from which the unit cost, based on \$340/ton caustic, is:

$$(0.0206)(0.17) = \$0.00350/\text{gal leachate}$$

or:

$$0.0035 \times 1000 = \$3.50/1,000 \text{ gal}$$

The preceding calculations are combined in equation 10, which can be used to estimate the daily NaOH usage:

$$60 \times Q \times T \times [0.334 \times 10^{-9H} + 1.2 \times 10^{-5} \times (Fe)], \text{ lb NaOH/day (10)}$$

where: Q = leachate flow, gpm

T = operating hours/day

Fe = iron, mg/L

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### 3.2 Oxygen Consumption

The oxygen demand is calculated by converting the leachate iron from mg/L to lbs/gal:

$$\frac{(1,687)(3.785)}{(1,000)(453.6)} = 0.014 \text{ lbs Fe/gal}$$

and noting that 1 lb of oxygen oxidizes 7 lbs of Fe:

$$\frac{0.014}{7} = 0.002 \text{ lbs O}_2/\text{gal.}$$

The amount of oxygen required is:

$$(0.002)(230)(60)(20) = 555 \text{ lbs/day}$$

Two aerators were used to deliver oxygen to the reactor for the data given in Table 1. Each is rated to transfer 3 lb O<sub>2</sub>/HP-hr to the solution as dissolved oxygen, or:

$$(2)(3)(7.5)(20) = 900 \text{ lbs/day}$$

which is in excess of that required by calculation. However, one aerator would not have provided the necessary oxygen demand.

Equation 11 permits the daily oxygen requirement to be estimated:

$$7.15 \cdot 10^{-5} \cdot (Fe) \cdot Q \cdot T, \text{ lbs O}_2/\text{day} \quad (11)$$

The daily cost of providing this oxygen, assuming an Aeration Industry aerator is used, is approximately:

$$1.8 \cdot 10^{-5} \cdot (Fe) \cdot Q \cdot T \cdot C, \text{ $/day} \quad (12)$$

where C = \$/kWhr

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For the data in this example, the cost to operate the aerator was:

$$7.5 \text{ (HP)} \cdot 2 \cdot 0.746 \text{ (kW/HP)} \cdot 20 \text{ (Hr)} \cdot \$0.055/\text{kWhr} = \$12/\text{day}$$

or:

$$\frac{\$12}{230 \cdot 60 \cdot 20} = \$0.000045/\text{gal} = \$0.045/1,000 \text{ gal}$$

### 3.3 Flocculant Usage

Flocculant usage is calculated from the make-up strength, lbs/gal, and the addition rate. From Table 1, the addition to the rotary thickener was

$$0.019 \text{ (lb/gal)} \cdot 4 \text{ gpm} \cdot 60 \text{ (min/hr)} \cdot 20 \text{ hr} = 91.2 \text{ lbs/day}$$

and to the belt filter press:

$$0.01 \text{ (lb/gal)} \cdot 2 \text{ gpm} \cdot 60 \text{ (min/hr)} \cdot 20 \text{ hr} = 24.0 \text{ lbs/day}$$

giving a total usage of 115 lbs/day. Nearly 80% of the flocculant is used to prepare the feed to the rotary sludge thickener.

The flocculant used at Keystone was Percol 730 (Allied-Colloid) which currently sells for \$3.15/lb in small lots. Using this figure gives the daily cost as:

$$115 \cdot 3.15 = \$362/\text{day}$$

The unit cost is:

$$\frac{\$362}{230 \cdot 60 \cdot 20} = \$0.0013/\text{gal} = \$1.31/1,000 \text{ gal}$$

P005633

### 3.4 Sludge Load

The sludge load can be estimated from the iron and suspended solids content of the leachate by converting the concentrations in mg/L to pounds/gal:

$$8.34 \times 10^{-6} \times [1.91 \times (\text{Fe}) = \text{TSS}], \text{ lbs solids/gal} \quad (13)$$

where: TSS = total suspended solids, mg/L

Using the information in Table 1, the calculated solids load in pounds/day:

$$8.34 \times 10^{-6} \times (1.91 \times 1,687 = 1870) \times 230 \times 60 \times 20 = 11,727 \text{ lbs/day}$$

This should be increased to allow for aluminum and manganese hydroxides:

$$1.15 \times 11,727 = 13,500 \text{ lbs solids/day}$$

or:

$$\frac{13,500}{230 \times 60 \times 20} = 0.049 \text{ lbs solids/gal}$$

### 3.5 Floc Factor

Floc usage is usually related to the amount of sludge produced. By ratioing these two quantities a factor may be developed for estimating floc usage and cost. This factor should be developed from historical data. Using the above data gives:

$$\text{Ratio} = \frac{115 \text{ lb floc/day}}{13,500 \text{ lb sludge/day}} = 0.0085 \text{ lb floc/lb sludge}$$

which is increased 25 percent to allow for upsets:

$$\text{Floc Factor} = 1.25 \times 0.0085 = 0.011 \text{ lb floc/lb sludge}$$

P005634

Relative to usage in the mining industry, flocculant usage at Keystone is very high. Typical usage in mining is 0.00005 lbs floc/lb solids (i.e., 0.1 lb/ton).

### 3.6 Operating Costs

Operating costs consist of labor, reagents, power, and maintenance. These are estimated in Table 2:

Table 2. Estimated Operating Costs  
for January 28, 1991

Parameters	\$ / day	%
Power: 40 HP * 0.746 * 24 * \$0.055/kWhr	39	2.1
NaOH: 5,700 lbs/day * \$0.17/lb	969	52.7
Floc: 125 lbs/day * \$3.15/lb	394	21.4
Labor: 20 man-hr/day * \$14.40/hr	288	15.7
Maintenance & Supplies:	150	8.1
<b>Total</b>	<b>\$1,840</b>	<b>100.0</b>
1000 Gal/day	267	
\$/1000 gal	\$6.67	

This estimate shows that reagents comprise nearly 75% of the total operating cost.

P005635

## 4 SYSTEM ANALYSIS

The CST system has certain advantages and disadvantages when compared to lime-based system. These are presented in Tables 3 and 4, respectively.

Table 3. System Advantages

- (1) Efficient utilization of reactants
- (2) Rapid reaction rates
- (3) Precise control of reaction pH
- (4) High metal precipitation efficiency
- (5) Small sludge volume
- (6) No sludge recycle
- (7) Lower plant capital cost
- (8) Possibility of remote process control
- (9) Portability

The system advantages listed in Table 3 are clearly selling features that can be used to balance the main disadvantage of high reagent costs.

Table 4. System Disadvantages

- (1) High reagent costs
- (2) Does not precipitate sulfates
- (3) Adds sodium to the effluent
- (4) Requires at least two solids recovery steps

P005636

Table 5 provides a comparison between the operating cost of a lime-based plant and a CST system.

Table 5. Operating Cost Comparison

<u>Parameters</u>	<u>\$/day</u>	
	<u>NaOH Based</u>	<u>Lime-Based</u>
Alkali	2,100	600
Floc	860	360
Labor	290	580
Power	70	130
<u>Maintenance &amp; Supplies</u>	<u>200</u>	<u>200</u>
<b>Total</b>	<b>3,520</b>	<b>1,870</b>
 1,000 gal/day	600	600
 \$/1,000 gal	5.87	3.12
 <u>Assumptions:</u>		
Connected HP	85	130
Labor, man-hr/day	20	40
NaOH/Lime usage	3.5	
Alkali cost, \$/ton	340	80
Floc usage, relative	2	1

This operation cost comparison indicates that an additional \$1,650/day is required to operate the NaOH-based system.

Over a year, this difference amounts to nearly \$600,000. If the two plants are operated for 10 years, the NaOH-based system must be constructed for about \$6,000,000 less than the lime-based system to break even.

If the initial difference in plant capital is considered, and the future value of this difference over 10 years is greater than the accumulative difference in yearly operating costs, the NaOH-based system would be favored.

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Table 6 gives a capital cost comparison for the two systems based on a flow of 500 gpm. Only the major equipment items are considered and it is assumed that the balance of the plant would be similar for the two systems.

The NaOH-based system costs are based on a detailed cost estimate presented later in this report. Those for the lime-based system are estimates made by the writer based on the HDS flowsheet. The NaOH-based costs are installed cost; the lime-based costs are delivered equipment cost. The delivered cost were converted to installed cost using a 1.65 multiplier (this was the ratio of installed-to-bare equipment cost found for the NaOH-based equipment).

Table 6. Capital Cost Comparison

<u>Equipment</u>	\$ / day	
	NaOH-Based	Lime-Based
Feed pump	6,000	2,450
Main reactor	5,810	55,000
Aerator	10,950	
Air compressor		20,000
Reactor disc pump	9,000	11,250
NaOH tank, pump, and flowmeter	13,835	
Lime silo and slaker		35,000
Lime reactor		25,000
Clarifier/thickener	110,700	105,200
Sludge w/f pump	6,920	28,500
Sludge recycle pump		16,500
Belt filter press	110,020	
Floc tanks, mixers, pumps	29,000	14,500
Conveyer	11,500	
<u>Effluent pump</u>	<u>21,000</u>	<u>3,600</u>
<u>Total</u>	<u>334,735</u>	<u>317,000</u>
Installed cost	334,735	523,050

Assumptions:

- (1) Installed cost = delivered equipment cost \* 1.65.
- (2) The lime-based system reactors are steel tanks.

0005638

(3). Lime reactor sized for 2 hr retention time.

Table 6 indicates a difference of \$188,315 in favor of the NaOH-based system. A detailed capital cost estimate should be performed to obtain a more reliable comparison.

A high cost burden for the NaOH-based system is the belt filter press, which is not required by the lime-based system, and which adds about \$110,000 to the equipment cost (see Table 3, Item 4).

The writer feels that a detailed capital cost analysis for a lime-based system will show costs comparable to those developed for the NaOH-based system. The \$11 million expenditure for the Conemaugh lime treatment plant seems extreme and may be based on extraordinary circumstances not usually encountered in practice.

The writer's experience tells him that given two systems, both capable of achieving the same end, the one costing less will usually be selected by the majority of clients requiring such systems.

The NaOH-based system has two principal disadvantages:

- (1) high price of caustic and
- (2) high flocculant consumption.

Sodium hydroxide is a by-product of chlorine manufacturing. Chlorine usage is decreasing, resulting in a decrease in the production of sodium hydroxide. However, demand for caustic is not decreasing, which is causing its price to increase. Long-term prospects for lower caustic prices are not good.

The writer believes that high flocculant usage is brought about by using sodium hydroxide. Sodium ions tend to peptize hydroxides, which requires higher polymer additions to overcome this effect. When lime is used, the calcium ion acts as a coagulant to neutralize surface charges, which reduces flocculant usage. This does not happen when sodium hydroxide is used.

P005639

The writer also believes that the rotary sludge thickener requires higher flocculant usage to ensure that a strong aggregate is produced ahead of the unit. I don't believe a delicate floc (i.e., one that is produced at lower floc additions) would be recovered at high efficiency in the rotary thickener. Gravity thickeners are less demanding in terms of flocculant addition.

Mr. John Anderson indicated to the writer that the rotary sludge thickener works best if the feed contains 2 to 3 percent solids by weight (instead of 0.5 to 1 percent). It is unlikely that coal refuse leachates would ever contain enough iron to give this solids concentration. Mr. Anderson indicated that he is thinking about a flotation process that would preconcentrate the precipitants before the sludge thickener. If this comes about, the CST process would involve three (3) solids concentration steps!

The CST system is unique in that it brings oxygen, alkali, and leachate together in a way that results in a fast N-O-P (neutralization-oxidation-precipitation) reaction. In this regard, it beats lime-based systems hands down.

When the CST system was first developed (1987), sodium hydroxide was much cheaper than it is today. Because caustic costs are unlikely to decrease, CST, Inc. should investigate using mixtures of sodium hydroxide and slaked lime to reduce costs. For example, if caustic usage can be reduced 50 percent, the difference being made up by lime, total alkali cost would decrease nearly 30 percent, from \$2,100 to \$1,500/day (see Table 5).

This type of development work would require the construction of a small CST reactor system. For example, a 1 HP Aeration Industries aerator would oxidize 21 lbs of Fe per hour. If the leachate contains 2,000 mg Fe/L, the leachate feed rate to consume this amount of oxygen is limited to about 20 gpm. A 200 gallon reactor (2.5' diam. x 5') would handle this flow and provide 10 minutes retention time. Of course, if the iron content decreases, the flow can be increased. If this suggestion is followed, provision should be made for recycling part of the reactor discharge to the feed.

The writer feels that floc usage can be reduced by replacing the rotary sludge thickener with a gravity sedimentation device; e.g., high-capacity thickeners, solids-contact clarifiers, lamella thickeners, Dorr-Oliver flocculators, etc. As pointed out

Later, the use of a rotary sludge thickener becomes impractical when the leachate flow rate exceeds 500 gpm.

## 5. DOCUMENTATION

A capital cost estimate is broken down into five cost categories:

- (1) mechanical equipment
- (2) instrumentation
- (3) electrical
- (4) civil and structural
- (5) summary

### 5.1 Cost Estimate Basis

Budget costs were obtained from equipment manufacturers for the major equipment items; i.e., for pumps, tanks, mixers, flowmeters, a rotary sludge thickener, a belt filter press, and a high-capacity thickener. Equipment costs were increased 4 percent to allow for freight. Instrumentation and electrical costs were provided by POWER Engineers.

The installed equipment cost was arrived at by determining:

- (1) labor costs for installing the bare equipment
- (2) cost of materials needed to complete the installation (normally piping, valves, fittings)
- (3) labor costs for installing the materials, and
- (4) subcontract work (earthwork, concrete, etc.).

A labor rate of \$34/hr was used in calculating labor costs. POWER Engineers followed a similar method for arriving at installed costs.

The building cost was obtained from GARCO (Spokane) and is quoted erected. Earthwork, concrete, lighting, structural steel, carpentry, etc., was estimated from data in the 1989 and 1990 Means construction guide reference books.

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It was assumed that CST, Inc., would be its own construction manager, and no allowance was made for construction management and fees other than those shown in the estimate.

Engineering costs were based on 12 drawings, each at \$2,500. Working capital of \$42,000 was allotted for start-up and no contingency was allowed.

Appendix A contains the detailed cost breakdown for the 500 gpm base case. Appendix B contains a summary of all four cases, but reports only the installed costs.

Appendix C contains the system flowsheet and equipment arrangement drawings defined as follows:

DWG G1	Raw Water-Solids Management System P&ID Flowsheet: for rotary sludge thickener
DWG G2	Building Plan and Sections: for 4' dia x 24' RST
DWG G3	Building Plan and Sections: for 5' dia x 24' RST
DWG G4	Raw Water - Solids Management System P & ID Flowsheet: for gravity thickener/clarifier
DWG G5	Building Plan and Sections: for thickener/clarifier

## 5.2 P&ID Flowsheets

Flowsheets G1 and G3 are identical except that G1 shows a rotary sludge thickener, while in G3, this is replaced by a conventional high-capacity thickener-clarifier. Flowsheet G1 is essentially identical to the Keystone plant flowsheet.

Run-off from the refuse piles (or coal stockpiles, etc.) is stored in a 2 million gallon equalization pond (270 ft L x 100 ft W x 8 ft D). The bottom of the pond is lined with compacted clay or fine shale. A synthetic membrane liner (40 mil HDPE) might be required which would add about \$35,000 to the cost estimate.

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Leachate is pumped from the pond by a 500 gpm fixed speed submersible pump. A manual bypass valve on the pump discharge can be set to recycle from 0 to 300 gpm back to the pond, thus giving the plant an adjustable feed rate of 200 to 500 gpm.

Two level switches interlock with the pump. The low-level switch turns the pump off; the high-level switch turns the pump on.

Both the leachate flowrate and reactor pH are measured and signals are sent to the PLC.

The discharge from the reactor is pumped to the rotary sludge thickener (RST) by a variable-speed pump. This pump could be eliminated if the CST reactor is elevated sufficiently high to permit gravity flow to the RST. This option was not pursued.

A level sensor in the CST reactor and a controller vary the pump speed to maintain a balance between output and input. The pump suction is connected directly to the reactor. To permit this, the reactor should be extended 2 feet and an internal weir installed to provide an isolated sump.

Caustic is metered to the reactor using a variable-speed gear pump which is controlled by the PLC. A gear pump is cheaper than a two-stage piston pump used at Keystone. A caustic flowmeter and recorder have been added for accounting purposes.

Mr. Anderson indicated to the writer that a 5' diameter by 24' long rotary sludge thickener would be required to handle a flow of 500 gpm. The cost of this unit is \$180,000. A thickener to handle the same flow would be 26' in diameter and would cost \$88,000 (Envir-O-Clear Corp.).

To compare these two options, their costs are expressed in \$/sq ft. For the rotary sludge thickener the cost is \$180,000/377, or \$477/sq ft; for the thickener, the cost is \$88,000/531, or \$166/sq ft.

This comparison shows, all things being equal, that it is more economic to use a high-capacity thickener than a rotary sludge thickener. Clearly, for throughputs over 500 gpm it becomes uneconomic to use a RST.

P005643

Flowsneet G4 shows a high-capacity thickener installed in place of the rotary sludge thickener. In either case, thickener sludge is pumped to the belt filter press (BFP) using a variable speed pump. The pumping rate is measured by a flowmeter, either magnetic or ultrasonic.

The belt filter press discharge is fed to a belt conveyor for off-loading onto a truck. Filtrate from the BFP and solution from either type of thickener is sent to the treated water pond, where it is pumped to the watershed.

The treated water pond was sized to store only 500,000 gallons. The writer could see no good reason for sizing the pond to contain the same volume as the equalization pond. In fact, the volume could be reduced to several hundred thousand gallons.

Provision should be made in the pond for collecting and removing tramp solids which discharge with the clean solutions. The pond also supplies water to the plant for wash-down, sprays, floc make-up, etc., via a small, high-pressure submersible pump.

The plant contains a small vertical sump pump which discharges into the equalization pond.

Flocculant is made up in two sets of tanks, each set consisting of two 600 gallons polyethylene tanks equipped with gear-driven, tank-mounted mixers. Two variable-speed rotary cavity pumps are used to deliver the flocculant to the thickener and belt filter press. Each floc line is monitored with a flowmeter.

A general material balance is shown on each P&ID drawing.

The total connected (installed) power is 103 HP. Of this, 30 HP is for the treated water discharge pump. This pump was sized to deliver 600 gpm through a 6" 2,000 ft line against a head of 100 ft.

It was visualized that the plant would be run from a small control panel, although local start/stop stations would be available at each motor. The PLC and MCC would be contained in separate housings.

8005644

### 5.3 General Arrangement Drawings

A 44' x 40' 6" building was selected as being the smallest building to provide adequate room for containing the equipment in flowsheet G1. Drawings G2 and G3 show the layout for the 4' x 12' RST and G3 shows the arrangement for the 5' x 25' RST. Both the RST and BFP are elevated off the floor with separate operating floors for each piece of equipment. In G3, the laboratory-office and bathroom area were sacrificed to accommodate the larger RST.

Drawing G5 shows how the equipment is arranged when a thickener replaces the RST. The building size has been reduced to 40' x 36' x 14' 6", while still accommodating a small lab/office, bathroom, and electrical area.

### 5.4 Cost Summaries

The capital cost estimate for the 500 gpm base case is abstracted from Appendix A in Table 7.

P005645

Table 7 Capital Cost for 500 gpm Plant

<u>Installation, \$</u>						
<u>Area</u>	<u>Equd</u>	<u>Md</u>	<u>Labor</u>	<u>Sub-C</u>	<u>Total</u>	<u>Total</u>
Equipment	364,403	31,122	28,790	8,680	68,592	432,995
Instruments	15,056	4,000	12,100	12,100	16,100	31,156
Electrical	36,815	3,200	15,025	21,855	640,080	76,895
Civil-Mech	0	2,150	4,850	93,980	113,980	145,980
Subtotal	416,274	40,274	60,765	136,615	238,752	687,027
As %	100	10	15	33	57	165
CST O & A						14,500
Engineering						30,000
Licenses						2,500
Insurance						5,000
Contingency						0
Working Cap						42,000
Subtotal						94,000
As %						23
Project						781,027
As %						188

The costs for the other three cases are summarized in Table 8. The portable plant does not involve any solids-liquid separation equipment. The other cases use gravity thickeners for the first solids concentration step.

Table 8. Capital Cost for Four Plant Sizes (HCTC)

<u>Description/Unit</u>	INSTALLED COST, \$			
	<u>Portable</u>	<u>500</u>	<u>1,000</u>	<u>2,000</u>
Mechanical Equipment	\$ 45,601	\$ 350,253	\$ 530,828	\$ 807,648
Instrumentation	18,656	31,156	33,326	36,801
Electrical	18,140	76,895	114,060	169,705
Civil & Mechanical	<u>1,000</u>	<u>145,980</u>	<u>189,080</u>	<u>235,390</u>
SUB-TOTAL	\$ 83,397	\$ 604,284	\$ 887,294	\$ 1,249,334
Other Costs (*)	<u>41,500</u>	<u>94,000</u>	<u>148,500</u>	<u>243,500</u>
TOTAL	\$ 124,897	\$ 698,284	\$ 1,015,794	\$ 1,492,844
Capital Cost per Gallon	250	1,397	1,016	746

(\*) includes CST O&A, engineering, licenses, insurance, legal, working capital.

## 6. SUMMARY AND CONCLUSIONS

The CST NaOH-based wastewater treatment system is an alternative to lime-based systems treating acid leachates derived from coal refuse piles, coal stockpiles, and mine waste dumps.

The CST system has advantages over lime-based system which include (1) rapid reaction rates, (2) efficient utilization of reactants, (3) complete precipitation of iron without sludge recycle, (4) smaller sludge volume, (5) lower plant capital costs, and (6) portability.

It has three disadvantages: (1) high price of caustic, (2) high flocculant consumption, and (3) the system requires two stages of solids dewatering.

P005647

The first two factors result in operating costs that are approximately 1.8 times the operating cost of a lime-based system. The third factor markedly increases the plant capital cost.

One way to reduce the flocculant usage and cost, as well as plant capital cost, is to replace the rotary sludge thickener with a conventional gravity thickener that is designed for waste-water treatment.

There are a number of workable choices. Gravity settling is less vigorous than the action occurring in the rotary sludge thickener. The floc structure need not be as strong, which means less flocculant is required to settle the solids.

Operating costs can also be reduced by replacing some of the sodium hydroxide with slaked lime. For example, replacing half of the caustic with lime can lower neutralizing costs 30 percent.

Employing lime will precipitate gypsum, thus lowering the sulfate level. And, it should help coagulate the precipitates and lower flocculant usage. However, it may also lower the reaction rates.

To determine the merits of this approach, it is recommended that a small, portable 20 to 30 gal/min CST reactor, utilizing a 1 HP aerator, be constructed to investigate replacing some of the caustic with slaked lime.

Experimentation with this unit at the Keystone site should:

- (1) reveal whether a NaOH-lime based system is feasible
- (2) identify significant operating problems
- (3) identify to what extent it can reduce reagent costs over a NaOH-based system.

The writer believes that aggressive development work is required to utilize the advantages offered by the CST reactor in developing business applications for treating acidic leachates.

2005648

A COST BREAKDOWN - 500 GPM CASE

P005649

POWER Engineer

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE; 600 gpm

Description	Equipment	Equipment				Installation				Total		
		Inv Nbr	Vendor	Inv Qty	Unit Cost	Inv Unit Total	Matl	Labor	Sub-Cont			
<b>MECHANICAL EQUIPMENT</b>												
Pump, 600 gpm, 30 ft. TDH, 260' elevation type, w/motor	1000 Grundfos	16	1	60	16	2,365	2,449	1,600	1,450	300	3,450	5,339
Reactor, 2000 gal, 6'-0" x 14' modified for internal pump	1010 CAR	1	1	60	4,000	4,160	1,650	1,650	1,650	350	10,350	
Aerator, 3 ft. dia/4 ft. w/motor	1020 Aeration Unit	7.5	2	60	5,000	10,400	250	300				
Pump, 600 gpm, 15 ft. TDH, w/ V-belt drive, VFD motor, rubber-lined parts	1030 Varimac	6	1	60	6,000	9,240	460	1,900	400	2,160	9,004	
Tank, 6000 gal, 8'-0" x 14' CS, to hold 50% NaCl at 65 F, heat-exch and skid-mtd, w/RJ10 translation, parts	1040 Vapex Tank	1	1	60	4,350	4,524	650	1,600	600	2,860	7,114	
Pump, gear, 55 x 16 ton, mach oval, 1" pipe, 0-25 gpm 600 head, w/VFD motor, base, coupling	1050 Line-flow	2	1	60	2	3,347	3,614	650	650	1,200	4,114	
Flowmeter, 0-20 gpm, 1" SS, 60% NaCl service, w/adjust. to recorder & accumulator	1060	1	1	60	1,200	1,240	100	600	600	100	1,844	
Rotary sludge thickener (RST).												
1 ton sludge (dry basis), 800 gpm hydraulic, 8'0" x 24', c/w Grunder line and motor	1070 T11	3	1	60	3	180,000	185,400	2,000	2,700	3,340	4,040	493,440
Vat, flocculator, 3'-0" x 6', (Inv. #1070)	T11	1	1	60	1							
Slitter and drive, 0-30 rpm (Inv. #1070)	T11	1	1	60	1							
Slipop, 4'11" x 2'6" x 4'0", CS	1080	1	1	60	1,900	1,960	100	200	400	400	1,940	
Pump, 125 gpm, 15 ft. TDH, 3" slurry pipe, c/w V-belt drive, VFD motor, rubber-lined parts	1090 Sherman	2	1	60	2	4,491	4,671	400	1,400	400	2,720	4,371
Ball filter press, 1 meter, 125 gpm hydraulic load, 1 ton sludge (dry basis), w/motor, var-i-speed drive, flocculator, var-i-speed motor	1100 Sherman	4	1	60	4	91,012	95,000	6,062	6,440	2,640	14,242	110,022

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE: 600 gpm

P-2

Description	Equipment ID#	Vendor	1P Qty	Unit Unit Unit Unit Unit Unit	1P Total Total Total Total Total Total	Equipment			Installation			Total	
						Matl	Labor	Std. Cont.	Matl	Labor	Std. Cont.		
Screen feeder, 4" x 10', rubber lined screen and trough	1110		2	ea	2	2,412	2,608	600	600	600	600	3,630	
Belt conveyor, portable type, 15' x 30', w/ motor, cover	1120		5	ea	6	10,330	10,743	800	300	600	600	11,543	
Tank, 600 gal, polyethylene	1130		4	ea		1,385	5,637	1,000	600	1,000	1,000	3,437	
Mixer, gear drive, 1750 rpm, 5' SS shaft, for 1130,	1140	Lightnin	1,1	ea	4	2,104	10,916	400	600	1,000	1,000	11,816	
Filter, floe mixing, w/scraper	1150		4	ea		200	8.2	400	600	1,000	1,000	1,172	
Pump, rotary screen, 10 gpm, 300' up, 1-1/2" pipe, 10' TD, for floe pumping service	1160	Wynona	1	2	ea	2	1,952	4,062	500	1,800	2,400	2,400	8,412
Floometer, variable-area, 0-10 gpm, 1", for 300' up service	1170	Wynona	2	ea		115	655	200	600	600	600	1,425	
Pump, 100 gpm, for water service, 100 psi, submersible	1180	Grundfos	10	1	ea	10	1,140	1,766	1,200	850	800	2,660	4,310
Pump, subp, 100 gpm, 30' TD, water level switch, rubber per se	1190	Wynona	2	1	ea	3	3,108	3,681	800	1,100	2,000	2,000	8,452
Pump, 600 gpm, submersible type, subp 2,000' & 10 psi, 100' TD	1200	Grundfos	30	1	ea	3,325	3,671	13,000	3,400	400	16,300	20,111	
			103.4			364,403	21,122	28,700	6,660	66,382	42,395		
												Sub-Total	

11002221

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE: 600 GPM

Description	Equipment			Installation			Total					
	Alt	Vendor	IP	Unit	Total	Matl	Labor	Sub-Contract				
				Unit	Cost							
<b>ALTERNATIVES</b>												
To replace 1010: 28' dia x 1'												
Hi-Cap thickener/clarifier, w/ upside legs, storage annular, racks, motor/drive, br ledge, desaturation tank	Enviroclear	3	1	ea	3	86,000	86,000	3,000	16,200	4,800	22,100	140,700
To replace 1100: Pump, dia- phragm, microstrainer control, 4 gm, 68, w/motor	Hilton-Ray	0.6	2	ea	1	1,201	0,414	600	2,000	2,000	2,000	4,314

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE: 600 gpm

Description	Equipment	Equipment			Installation			Total
		Qty	Unit	Total	Unit	Total	Unit	
<b>LIQUIDATION</b>								
Liquid level sensor element, w/remote readout for 1040, including L.E., L.P., & LA	1 Lot	100	100	400	400	400	1,100	
Temperature sensor element, w/remote readout for 1040, including, L.P., & LA	1 Lot	360	360	300	300	300	930	
Magnetic flowmeter, 0-600 gpm, for 4" line, pl 2 service, 50 l.O. reactor feed, w/FTI	1 ea	2,000	2,000	400	400	400	2,400	
Ultrasonic flowmeter, 0-150 gpm, 3" line, 50 l.O. BFP feed	1 ea	1,200	1,200	600	600	600	1,440	
PI element, a ultrasonic probe, for 1010, w/analog output, incl liquid level sensor element, for 1010, w/LT to PLC	1 ea	1,300	1,300	600	600	600	1,300	
Treated water pond level switch, L.P.	1 ea	600	600	400	400	400	1,200	
Equalization pond level switch, L.P. a L.S.	1 ea	600	600	300	300	300	900	
Pump level switch, L.S.	1 ea	600	600	300	300	300	900	
Pen recorder for treated plant, model 511, plow, 1 pl	2 ea	1,200	600	600	600	600	2,400	
PLC: SLC 500 System, /w 32:32A with PID, analog I/O, 8 aux. rack, power supply, 120 vac input/output cards	1 Lot	2,350	2,350	1,000	1,000	1,000	3,440	
PLC Accessory: programmer, memo book, battery, isolater	1 Lot	100	100	1,362	1,362	1,362	0	1,362
PLC NEMA 4 enclosure	1 Lot	1,000	1,000	300	300	300	1,300	
Control panel	1 Lot	2,300	2,300	2,300	2,300	2,300	4,600	
Instrumentation wiring	1 Lot	1,500	1,500	4,000	4,000	4,000	5,500	
Sub-Total		15,714	4,300	12,100	12,100	12,100	41,114	

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE, 500 kW

Description	Type	Vendor	Wt.	Qty	Unit	Unit	Equipment			Installation		
							Unit	Total	Mat.	Sub-Cont.	Mat.	Total

pt. 6

8555004

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE, 500 BHP

p. 6

Description	Nor	Vendor	IP	Qty	Unit	IP Total	Equipment			Installation			Total
							Unit	Total	Matl	Labor	Sub-Cont	Total	\$
<b>ELECTRICAL</b>													
4-section motor control center with 225 amp main breaker, 1 size 111, 16 size 1 comb in- at icl starter and 6 feeder breakers	1	Lot			16,200	3,000						3,000	21,200
25 KVA, 480/120 V step-down transformer	1	Lot			800	400						400	1,200
Single phase 240/120 V, 24 ckt panel board w/225 amp main breaker	1	Lot			535	325						325	860
SCR drive for 1090, 5 IP	1	ea			2,865	2,865						700	3,565
SCR drive for 1050, 2 IP	1	ea			2,310	2,310						700	3,010
SCR drive for 1190, 2 IP	4	ea			2,310	9,240						2,800	12,040
VF drive for 1090, 5 IP	1	ea			2,865	2,865						700	3,565
Heat tracing and controls, for 1040, to maintain 50% caustic so in at 65 F for 32 F ambient, sp heat 0.77 BTU/10°F	1	Lot				800						1,200	1,200
Interior lighting: 3 watts/SF, 30 fluorescent fixtures @ with 4-40 watt tubes (industrial)	4.76	\$/SF										9,215	9,215
Exterior lighting: 400 w Na vapor lamps mt'd on 20' Al pole includes excavation, concrete base, compaction, all fixtures	6	ea			2,015							12,090	12,090
Convenience outlets	1	Lot										550	550
Wiring to all motors	1	Lot										8,400	8,400
<b>Sub-Total</b>					36,315	3,200	15,025	2,400	6,000			40,030	76,895

B005655

## APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE; 500 gpm

Description	Qty	Unit	Equipment			Installation			Total \$
			Unit	Total	Mtl	Sub-Con	Total		
<b>CIVIL &amp; STRUCTURAL</b>									
Pre-Fab building, 44'x44'x14', w/siding, insulation, man door									
(2), truck door (10' x 12')									
ventilator, 40 psf snow load, 100 mph wind load									
Trenching, 2'D x 2'W									
Strip Footing, incl forms, reinforcing, 3000 psi concrete									
Slab on grade, 44'x44'x10", incl grading, compacted gravel pad, 3500 psi concrete placed by chute, joints, forms, finishing									
Septic field & tank, 1000 gal precast tank, trenching field, excavation, gravel backfill									
Heating; use 6 kw infrared unit									
Structural steel, for operating floor support, stairs, 6x16 I-beam									
Floor grating									
Hand railing									
Misc (stairs, hangars, etc)									
Lab/office construction									
Plumbing									
MCC room, cinder block									
Painting									
Leachate holding pond, 2 million gal, 270'L x 100'W x 8'C									
Excavation									
Compaction									
3900 CY	500 cu								
24,300	1,250								

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE; 500 gpm

p. 6

Description	Qty	Vendor	IP	Qty	Unit	IP	Equipment			Installation			Total
							Unit	Total	Matl	Labor	Sub-Cont	Total	
Clay, delivered													
Clay liner, installed													
Treated water pond, 0.5 million gal, 80'L x 80'W x 8'H (factored estimate)	2476	CY											
Lab equipment	1	Lot											
Sub-Total							0	2,150	4,850	93,960	113,980	145,980	

APPENDIX A: CAPITAL COST ESTIMATE FOR BASE CASE: 300 year

P. 9

Description	Equipment			IP			Installation			Total	
	Mar	Vendor	IP	Qty	Unit	Total	Matl	Labor	Sub-Cont		
<b>SUMMARY OF COSTS</b>											
Mechanical Equipment				103.4		364,403	31,122	26,790	6,860	40,937	432,895
Instrumentation						16,068	4,000	12,400	12,400	16,100	31,168
Electrical						36,815	2,300	16,025	21,685	40,000	106,835
Civil & Mechanical						0	2,160	4,860	92,980	113,980	145,980
<b>Sub-Total</b>						410,274	40,472	60,765	139,415	236,762	607,027
As %						16.0	10	16	37	67	16.5
As \$/sqm											1,314
O & A (assume cost is 10% contractor)											14,863
Engineering Cost (102500)/sqm											20,311
Licenses											1,600
Insurance & Legal											6,000
Contingency											0
Working Capital (1 month operating costs)											42,000
Sub-Total											94,311
Total Project Cost											101,321
As \$/sqm											1,362

B-COST BREAKDOWN - ALL CASES

8005659

POWER engineer

APPENDIX E: CAPITAL COST ESTIMATE

Description	Capital Cost Estimate				
	Estimated	1000	10000	100000	
<b>PROCESS EQUIPMENT</b>					
reactor feed pump, submersible	91	5599	5999	3093	3732
CO <sub>2</sub> reactor, w/ internal sump	1	1811	1811	9001	10346
generator, 3 kw alternator, motor	19	19560	19360	14337	15153
reactor discharge pump	3	9006	9006	13630	20690
batch tank, 6000 gallon	1	7174	7174	6374	7482
sump metering pump	3	1714	1714	1143	10830
batch flowmeter, w/cutsout		1948	1948	1948	1948
scraper, w/ sludge thickener		12	12	12	12
thickener clarifier		10700	107700	104321	
sump box	1	1963	1963	1963	1963
sludge pump	1	6921	10490	15300	
belt filter press	1	110022	166762	252764	
screw feeder	1	3558	5393	8174	
belt conveyor	1	11343	17236	26519	
sludge storage tanks	1	7437	11272	17386	
sludge mixer	1	11816	17910	27146	
sludge mixing eductor, w/hooper	1	1832	2777	5380	
sludge pump, rotary screw	1	6462	9795	14845	
sludge flowmeter	1	1453	2205	3343	
spray water pump, submersible	1	4319	5545	9920	
sump pump	1	6467	9787	14834	
effluent pump, submersible	1	20171	33574	46340	
<b>Sub-Total</b>		<b>45601</b>	<b>350253</b>	<b>530828</b>	<b>807648</b>

P005660

## APPENDIX B: CAPITAL COST ESTIMATE

Description	Installed Cost (\$)			
	Portable	500	1,000	5,000
Instrumentation				
Flow meter 1/2", 1/4", 1/8", 1/16"	100	100	100	100
Flow tank 75, 750, 7500	600	600	600	600
Flow flowmeter	2400	2400	3640	3640
Sludge flowmeter	72	1800	2700	4125
Reactor pH & CHT	1600	1600	1600	1600
Reactor LT to PLC	14	1200	1200	1200
Treated water conc LSL	900	900	900	900
Flow meter conc LSL & 100%	1000	1000	1000	1000
Level level switch LS	72	300	300	300
Pen recorder	2400	2400	2400	2400
Programmable logic controller	3444	3444	3444	3444
PLC accessories	1362	1362	1362	1362
PLC NEMA 4 enclosure	1900	1900	1900	1900
Control panel	72	4500	4500	4500
Instrumentation wiring	1400	3500	3500	3500
Sub-Total	16656	31156	33326	36601

## APPENDIX B: CAPITAL COST ESTIMATE

S-1

Description	Cost	Quantity	Unit	Total
<b>ELECTRICAL</b>				
Power source center	6000	1	unit	6000
20000 ft. underground line	1000	1	unit	1000
Single phase 120/120 V, 24 amp panel board, w/breaker	130	1	unit	130
SCSI drive for P3 disc auto	72	1	unit	72
SCSI drive for NaOH auto	3010	1	unit	3010
SCSI drive for file auto	72	1	unit	72
Variable frequency drive for P3	2	1	unit	2
Heat tracing for valve tank	1000	1	unit	1000
Underfloor lighting	72	1	unit	72
Exterior lighting	4030	1	unit	4030
Convenience outlets	650	650	unit	650
wiring to all meters	2100	2400	unit	13000
<b>Sub-Total:</b>	<b>18140</b>	<b>76898</b>		<b>114060</b>
				<b>169705</b>

P005662

## APPENDIX B: CAPITAL COST ESTIMATE

6.4

		Installed Cost		
<u>Land</u>				
Portable	100	1,000	1,000	
<u>STRUCTURE</u>				
Mobile building, erected	na	30000	37500	37500
Building foundation, shoring	na	3140	3900	3900
Building, 1000-sq-ft	na	1700	19100	19100
Steel, field & site	na	2090	2090	2090
Roofing	na	3000	3000	3000
Building steel	na	10000	16500	16500
Motor grader	na	3200	4100	4100
Excavating	na	800	750	7500
Exc	000	1000	1000	4000
Lab/office construction	na	1500	1500	1600
Shoring	na	1000	1000	1000
Reb, beam, cinder block	na	1500	2000	2000
Painting	na	3000	3000	3000
Leachate holding pond	na	34800	51840	72600
Treated water pond	na	18000	19700	19900
Lab equipment	na	25000	25000	25000
<b>Sub-Total</b>		<b>1000</b>	<b>145980</b>	<b>189090</b>
				<b>235390</b>

B005663

APPENDIX B: CAPITAL COST ESTIMATE

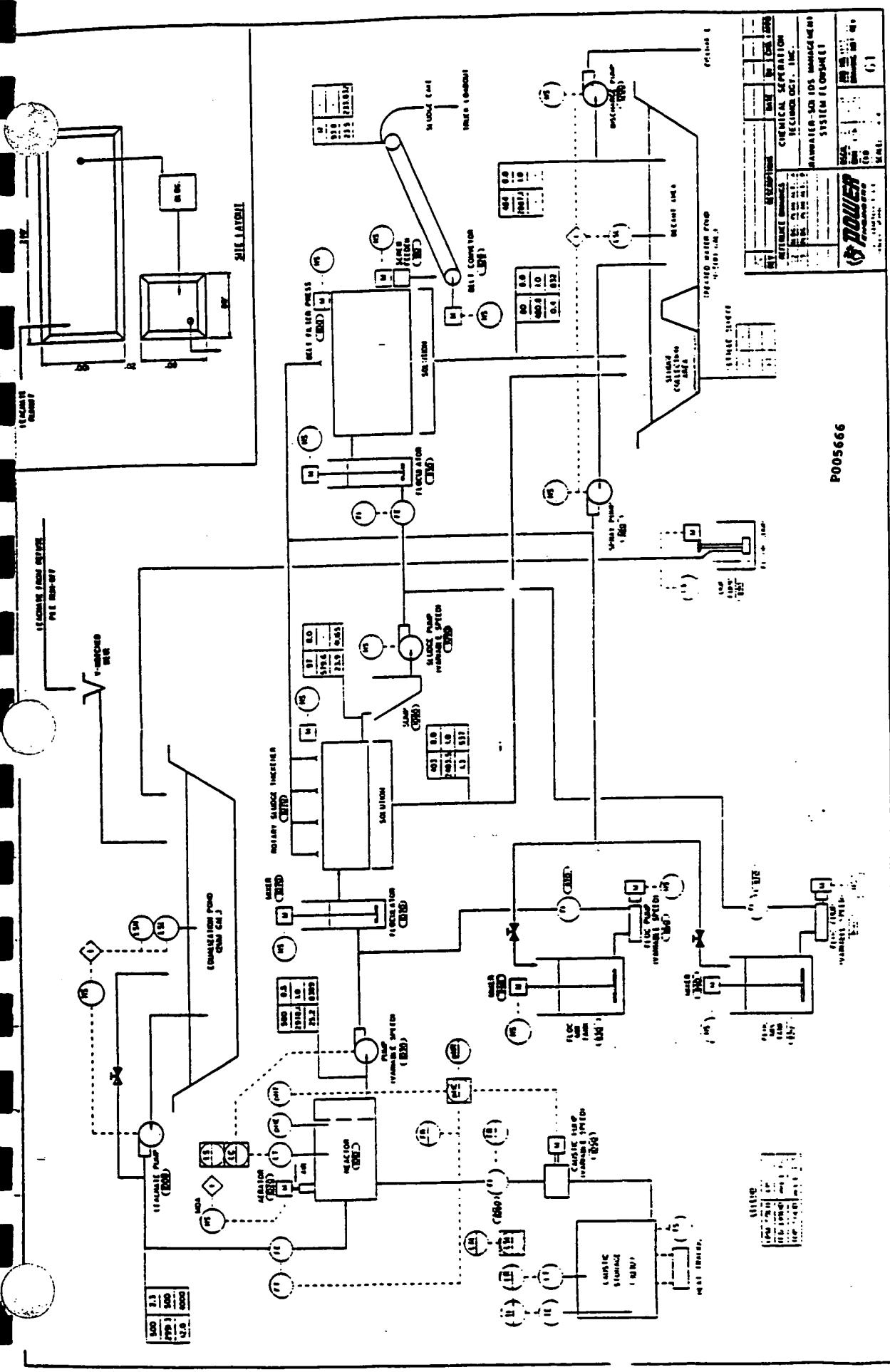
Description	Installed Costs			
	Portable	1,000	10,000	100,000
<b>Summarized Costs</b>				
Mechanical Equipment	4600	392000	100000	107448
Instrumentation	10000	31100	10000	33800
Electrical	13140	10395	14060	139705
Civil & Mechanical	1000	15980	100000	108360
Sub-Total	63397	604284	367294	1249344
As %/GPM	167	1000	367	323
GST & C.C.	5000	14500	22000	33000
Engineering	1000	30000	30000	30000
Consultants	50	50	5000	5000
Licenses	2500	2500	2500	2500
Insurance & Legal	2500	5000	5000	5000
Contingency	0	0	0	0
Working Capital	24000	42000	34000	168000
Sub-Total	41500	94000	142500	343844
Total Project Cost	104897	698284	1015794	1433844
As %/GPM	150	1367	1013	746

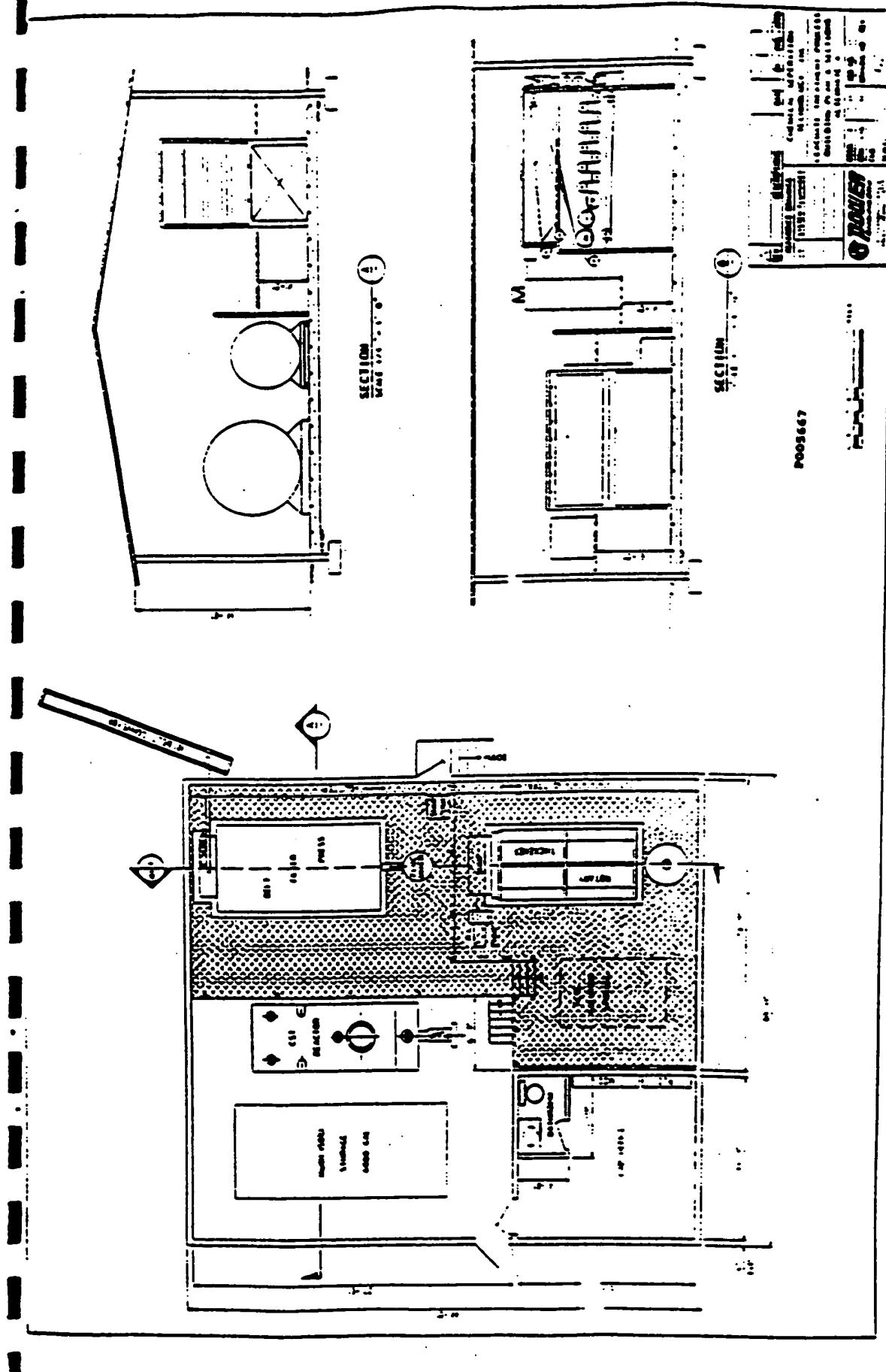
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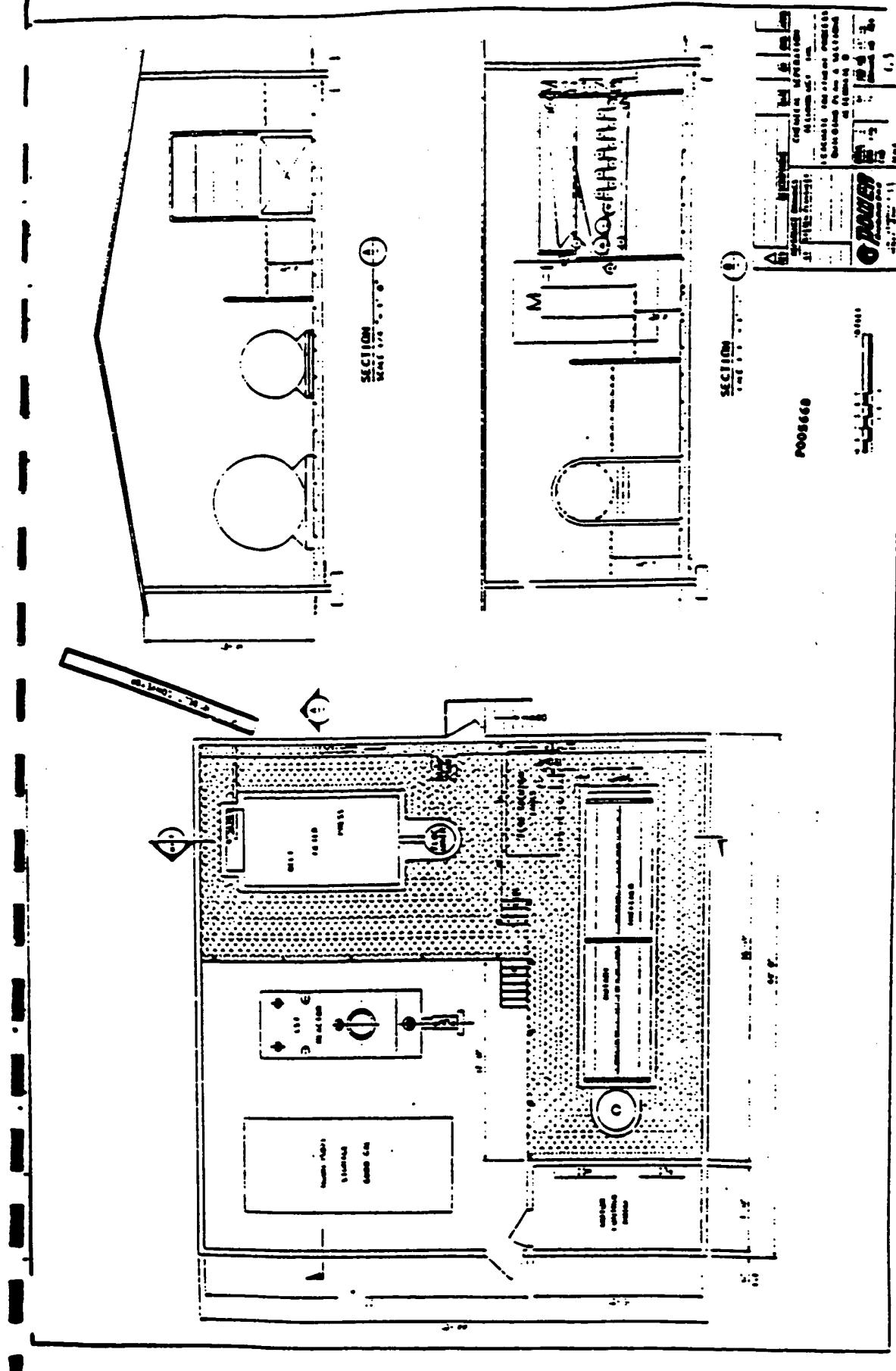
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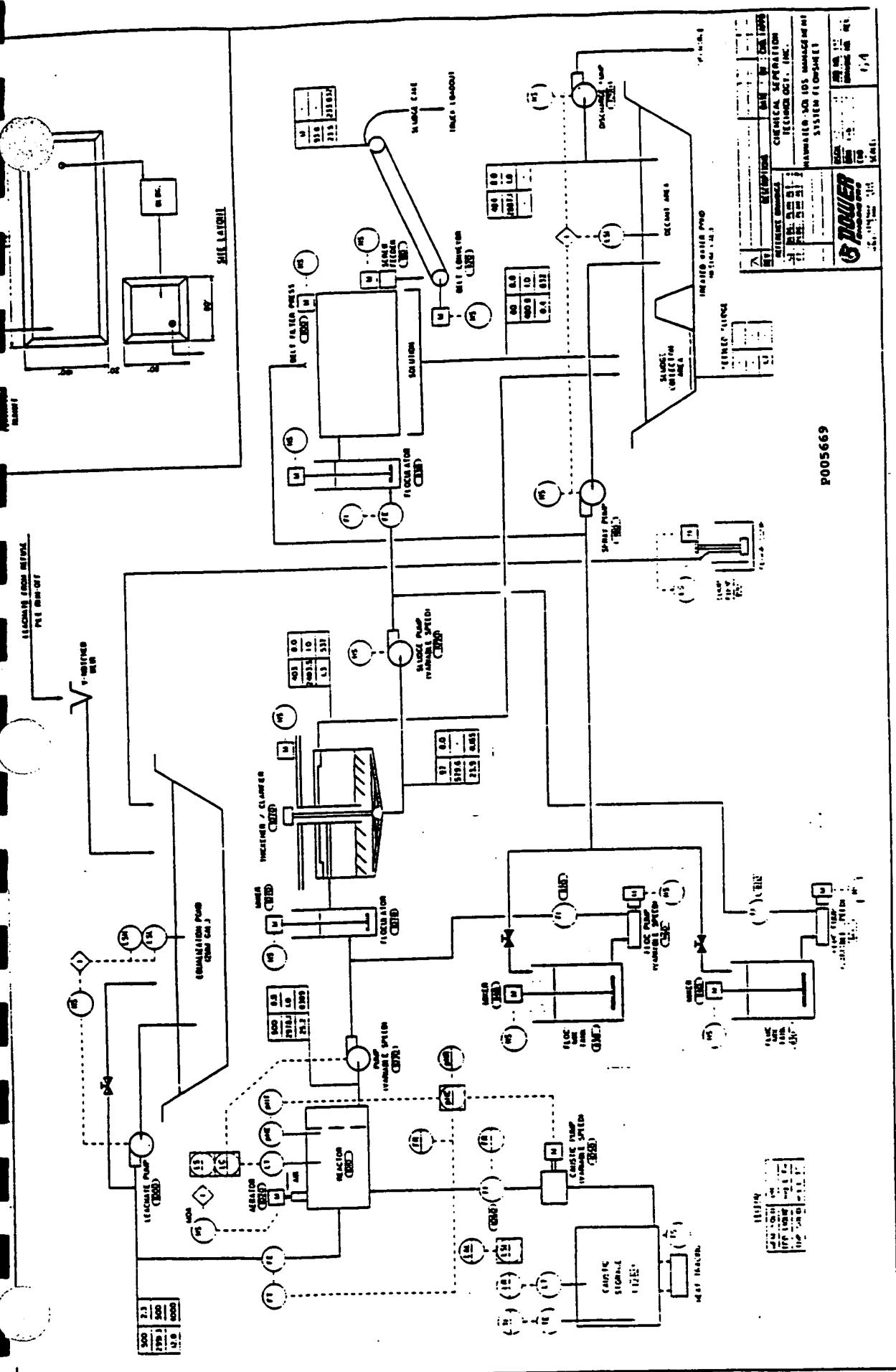
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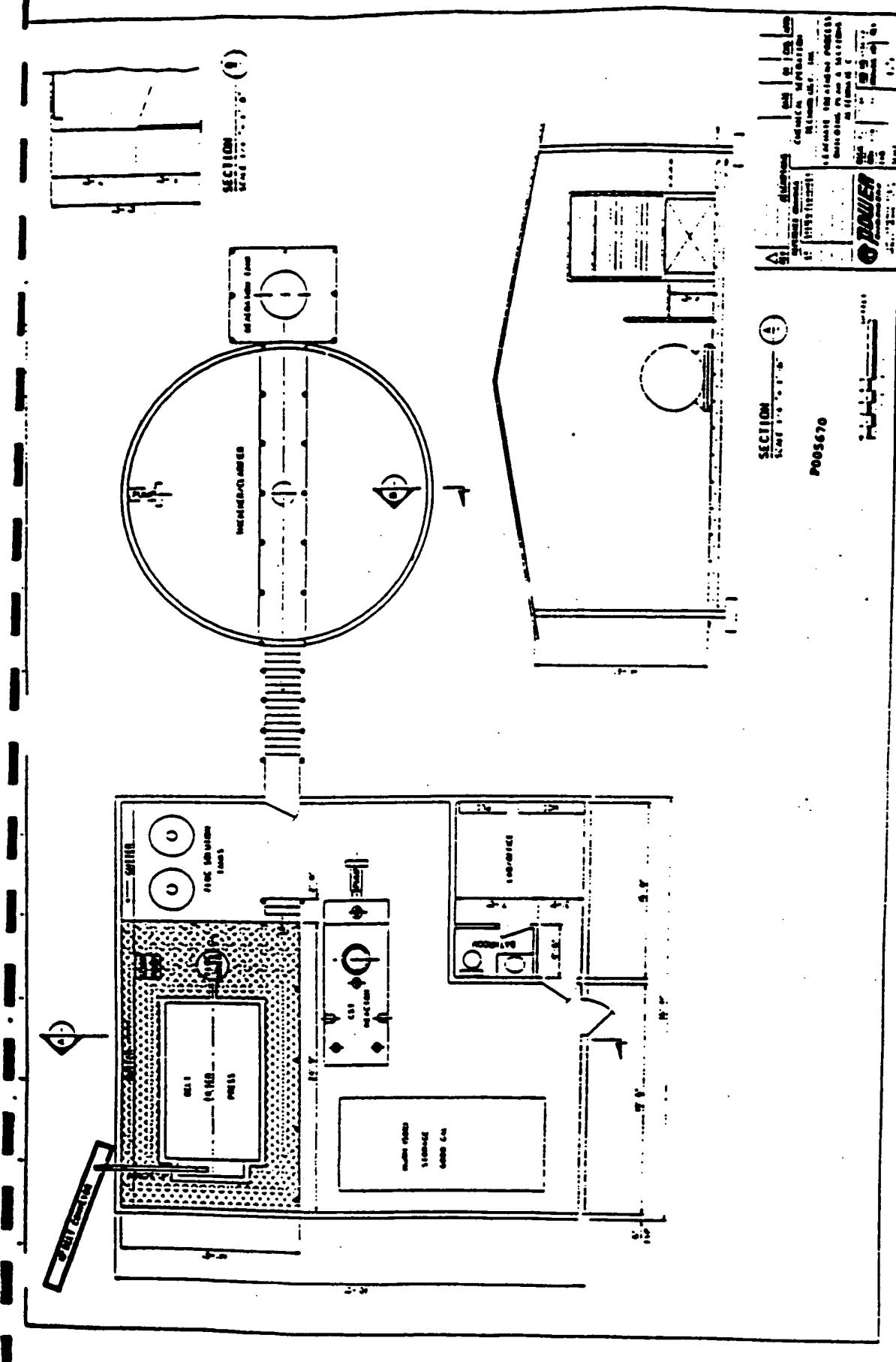
POWER Engine











**CHEMICAL SEPARATION TECHNOLOGY, INC.**

April 16, 1992

Summitville Consolidated Mining Co., Inc.  
P. O. Box 2G  
Del Norte, Colorado USA 81132

Attention Mr. Bill Williams

RE: Preliminary Proposal 4/14/92  
Reynolds ADIT

Dear Bill,

Per our telephone conversation 4/14/92 the effluent discharge quality at the above subject site should be as follows.

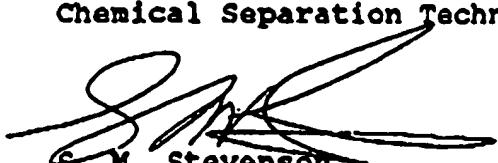
pH	7.5
Fe	< 1 Mg/l
Cu	< .1 Mg/l
Mn	< 1 Mg/l
Zn	< 1 Mg/l

All discharge limits have been determined from the raw water quality data that you forwarded to me earlier. Historically we have experienced metals discharge ranges in the .002 Mg/l ranges and those limits could be achievable at the ADIT site.

Please call me if I can be of further assistance.

Very truly yours,

Chemical Separation Technology, Inc.



S. M. Stevenson  
President

SMS/ds

201410

INNOVATIVE ENVIRONMENTAL TECHNOLOGIES  
Post Office Box 931 • McMurray, Pennsylvania 15317 • 412/942-0674 • FAX: 412/942-0536

0004006

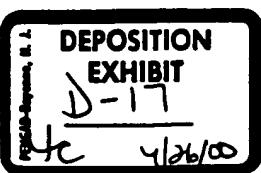
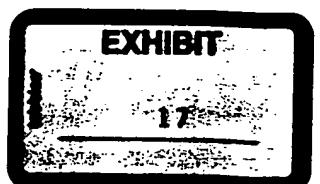
## IN THE UNITED STATES COURT OF FEDERAL CLAIMS

CHEMICAL SEPARATION TECHNOLOGY, INC. )  
and SANFORD M. STEVENSON, )  
Plaintiffs, )  
v. ) No. 97-21 C  
THE UNITED STATES, ) Judge Francis M. Allegra  
Defendant. )

**AFFIDAVIT OF SANFORD M. STEVENSON  
IN SUPPORT OF RESPONSE IN OPPOSITION TO  
DEFENDANT'S MOTION FOR PARTIAL SUMMARY JUDGMENT  
DECLARING U.S. PATENT NO. 5,370,800 INVALID**

And Now, this 21st day of May, 1999, I, Sanford M. Stevenson, being 57 years of age, of sound mind, and competent to testify, state under oath and penalty of perjury pursuant to 28 U.S.C. § 1746 the following upon my personal knowledge:

1. I am the president of Chemical Separation Technology, Inc. I was personally involved with the installation of the PITS System at the Summitville Mine for Summitville Consolidated Mining Company, Inc., (SCMCI) which is a subsidiary of Galactic Resources, Ltd. Construction of an operational PITS System was completed on August 5, 1992. I began initial startup of the installation of the PITS System at Summitville Mine on July 13, 1992 and left the site on July 24, 1992. I returned to the site on July 31, 1992 through August 2, 1992. (see Invoice attached as Exhibit 2) During this period of time, I was engaged in experimentation to finalize and make predictable the chemical process that ultimately became U.S. Patent No. 5,370,800, granted on December 6, 1994, entitled "Method for Removing Metal Compounds



from Waste Water" (hereinafter '800 Patent). ('800 Patent attached as Exhibit 3)

2. During the periods of time identified in paragraph 1 of this Affidavit, I calibrated and activated the reactor component of the PITS System which is disclosed in claims 1 through 9 of the Chemical Separation Technology, Inc., (CST) Patent No. 4,749,497, granted on June 7, 1988, entitled "Method and Apparatus for Treatment of Acidic Water" (hereinafter '497 Patent). ('497 Patent attached as Exhibit 4)

3. The PITS System as ultimately constructed and finalized on August 5, 1992, is the sludge dewatering components and chemistry, coupled with the reactor component covered by the '497 Patent, that falls within the scope of claims 1 through 25 of the Sanford M. Stevenson '800 Patent.

4. During the same period of time referenced in paragraph 1 of this Affidavit, the installation and activation of the sludge dewatering components and chemistry of the PITS System at the Summitville Mine were delayed because of control panel wiring problems, a lack of fresh water to mix and test polymers, as well as the testing and experimentation with the types, dosages, dilutions, and type specific injection and placement of polymers. The August 31, 1992, letter of SCMCI produced by the Government in response to Plaintiff's request for production of documents corroborates and articulates the experimentation process which CST and I underwent to finalize and ultimately reduce the method covered by the '800 Patent to practice. (letter attached as Exhibit 5). In addition, as late as August 2, 1992, after I left the site and was returning to Pittsburgh, the chemical process that became the method of the '800 patent was not finalized as is evidence by the fax of Mr. John Anderson to me wherein he details for me the problems with the sludge and states at the end, "Get the chemical treatment under control." (Fax

attached as Exhibit 6)

5. Prior to May 25, 1992, I had not reduced to practice the Method covered by the '800 patent, nor had I prepared any drawings which disclosed the type, dilution, dosage or injection placement of polymers that are disclosed by claims No. 21, 22, 23, of the '800 Patent. Prior to the experimentation during the period referenced in paragraph 1 of this Affidavit I was unsure that the method would work, and it was only around August 5, 1999 that the method had been refined to a degree of predictability. ✓

6. On April 14, 1992, I sent a preliminary budget proposal to SCMCI (letter attached as Exhibit 7). At the time that I sent such proposal, I was uncertain that the method which utilized the mechanical system embodied as the PITS System would function effectively. Mr. William B. Williams, of SCMCI, and I spoke about my uncertainty and I advised him that if the system didn't work, then SCMCI didn't have to pay for it. Mr. Williams memorialized my uncertainty in his purchase order of the system dated May 5, 1992, wherein it states "FINAL COSTS AND DETAILS TO BE VERIFIED BY PLANS AND SPECIFICATIONS TO FOLLOW". (Purchase Order attached as Exhibit 8)

7. Upon careful examination, none of the exhibits attached to Defendants Motion for Partial Summary Judgment discloses the process ultimately disclosed by the '800 Patent which is a method for removing metal compounds from waste water. The exhibits merely disclose the placement and sequential operational chain of the conventional water-treatment apparatus used to enable the chemical method disclosed in the '800 Patent.

8. Specifically, Exhibits 3, 10, 13 and 16, offered by Defendants in support of the fact that the method patented in the '800 Patent was reduced to practice, do not disclose the type,

injection placement or dilution of specific types of polymers claimed in claim numbers 21, 22, and 23 of the '800 Patent. Nor do any exhibits disclose the pH operational limits under the claims of the '497 Patent which make the '800 Patent method possible. Exhibit 10 attached to Defendant's Appendix are not copies of blueprints sent to SCMCI on May 14, 1992. The actual blueprint sent on that date is attached hereto as Exhibit 9.

9. Without disclosure of the information identified within paragraph 8 of this Affidavit, a skilled artisan could not reduce the method disclosed in the '800 patent to practice.

10. The method which is patented and disclosed by the '800 Patent was not reduced to practice until I completed experimentation with mixing, dilution, types and specific injection and placement of polymers and produced an effective and predictable method capable of patenting. The successful completion of such experimentation was accomplished on or about August 5, 1992.

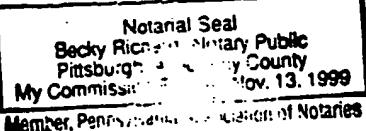


Sanford M. Stevenson

Sworn and Subscribed to me

this 21st day of May, 1999

Becky Richard  
Notary Public



45 Fed.Cl. 513  
53 U.S.P.Q.2d 1419  
(Cite as: 45 Fed.Cl. 513)  
<KeyCite Citations>

United States Court of Federal Claims.

**CHEMICAL SEPARATION TECHNOLOGY,  
INC., et al., Plaintiffs,  
v.  
The UNITED STATES, Defendant.**

**No. 97-21C.**

Dec. 14, 1999.

Holder of patent on method of treating industrial wastewater older brought suit against the United States, alleging infringement, and seeking just compensation for the government's unlicensed use of the patented technology. On defendant's motion for partial summary judgment, the Court of Federal Claims, Allegra, J., held that genuine issues of material fact as to whether method of treating industrial wastewater was ready for patenting prior to one year before patent application precluded summary judgment on issue whether "on sale bar" afforded defense to infringement claim.

Motion denied.

West Headnotes

**[1] Patents k76**  
291k76

Elements of the "on sale" defense to a patent infringement claim are: (1) that there was a definite commercial sale or offer for sale of the invention claimed in the patent more than one year before the patent application was filed; and (2) that at the time of that sale or offer for sale, the invention was ready for patenting. 35 U.S.C.A. § 102(b).

**[2] Federal Courts k1120**  
170Bk1120

Genuine issue of material fact as to whether method of treating industrial wastewater was ready for patenting prior to one year before patent application precluded summary judgment on issue whether "on sale bar" afforded defense to infringement claim. 35 U.S.C.A. § 102(b).

\*513 Louis M. Tarasi, Jr., Pittsburgh, Pennsylvania, for plaintiffs.

Cameron Eliot, U.S. Department of Justice, with whom was Acting Assistant Attorney General David W. Ogden.

## OPINION

ALLEGRA, Judge.

This patent infringement suit is before the court on defendant's motion for partial summary judgment. The defendant asks this court to declare U.S. Patent No. 5,370,800 invalid due to plaintiffs' alleged violation of \*514 the "on-sale bar" of 35 U.S.C. § 102(b)(1988). Following oral argument and after a careful review of the materials submitted by the parties, this court finds that the issue whether the plaintiffs violated the on-sale bar as to this patent presents material questions of fact and thus is not susceptible to summary judgment. Defendant's motion for partial summary judgment is therefore DENIED.

### I. Facts

The plaintiffs, Chemical Separation Technology, Inc. (CST), an Idaho corporation, and its president and major shareholder, S.M. Stevenson, are owners of a U.S. patent that relates to the treatment of waste water. The patent, U.S. Patent No. 5,370,800, (the 800 patent) was applied for on May 25, 1993, and granted on December 6, 1994. The patented "method" is used in an apparatus constructed by CST known as a "portable interim treatment system" (PIT System). The PIT System, using a series of chemical agents, precipitates hazardous minerals and compounds from industrial waste water, thereby cleaning the water. A critical feature of the "method," according to the plaintiffs, is the addition of cationic and anionic polymers [FN1] to the polluted water, thereby causing the minerals to clump together (flocculate) and producing water with metal ion concentrations below EPA limits.

FN1. A polymer is macromolecule, a string of organic or inorganic molecules, formed by the chemical union of at least five identical monomers, which are simple molecules or compounds usually made of carbon and exhibiting simple structure and low molecular weight. *See Hawley's Condensed*

*Chemical Dictionary* 900 (13th ed.1997). A cationic polymer exhibits a positive ionic charge, while an anionic polymer exhibits a negative ionic charge. *Id.* at 77, 223-24. Polymers aid in flocculation by forcing suspended metal particles to aggregate into clumps or tufts as they pass through a solution containing these long chains of inorganic or organic compounds. *Id.* at 506.

Prior to April 1992, CST had a number of prototype waste water treatment systems running involving physical plants very similar, if not identical, to the PIT System. At least two of these systems were being used in coal refuse landfills in Pennsylvania. An additional apparatus was in operation at the Pennsylvania Electric Company in Indiana, Pennsylvania, designed to precipitate iron, a ferrous metal, with the incidental removal of manganese and aluminum.

On April 14, 1992, CST made a formal offer of sale of a waste water treatment facility using the PIT System to Summitville Consolidated Mining Company ("Summitville"), a subsidiary of Galactic Resources, Inc., to remove copper from waste water produced at its gold mine in Summitville, Colorado. This offer was accepted by Summitville on April 24, 1992, and a purchase order was signed by an agent of Summitville on May 5, 1992. According to the plaintiffs, the system needed at the Summitville site was fundamentally different than that in use at the Indiana, Pennsylvania site, because the latter system was incapable of dealing with the copper-laden waste water produced at Summitville. In an effort to develop a polymer method that would deal with nonferrous pollutants such as copper, Mr. Stevenson allegedly engaged in an additional round of experimentation that was not completed until August 5, 1992. As such, plaintiffs allege that what they sold to Summitville in the spring of 1992 was an unfinished water treatment system, as the specific chemical process for removing non-ferrous metals, including the specific polymers, dilution, dosage or injection points to be used to effect the removal of non-ferrous minerals, was then incomplete.

Following the installation of the PIT System at Summitville, a number of different polymers were utilized, with varying degrees of success. Penny McPherson, the environmental manager at the Summitville mine, contacted the United States Department of Natural Resources and the Colorado Department of Health by letter of August 31, 1992, regarding the state of waste water treatment and copper removal. Her letter suggests that the specific

polymer mix was not finalized until August of 1992. [FN2]

FN2. Her letter reads, in pertinent part: [The PIT System was restarted] on July 27 ... and [Summitville] directly inject[ed] the ... polymer in the stream. Only moderate success was achieved with this direct injection method and the quality of the solution in the PITS tanks was not sufficient for discharge. It was evident a near-neutral pH water source would be needed to achieve proper Allied Colloids polymer dosages to the PITS and [Summitville] placed a pump and circulation pipeline in the clear solution (pH 7-8) near the top of the # 2 clarifier tank.

The Allied Colloids polymer was not compatible with this solution.... [Summitville] asked for technical assistance in solving the flocculation and sludge formation problems at the PITS... Once ... a polymer had been found that could be used with the PITS effluent ... the PITS performed very satisfactorily.

\*515 The PIT System was left in place at the Summitville site after Galactic abandoned the site. In December, 1992, defendant, through the EPA, began an emergency Superfund response action at the Summitville site. An outside firm, the Environmental Chemical Company (ECC), was retained to serve as the emergency response contractor to manage the clean-up of the site. The clean-up included treatment of waste water, which ECC accomplished by using the PIT System already installed at the site. In 1993, ECC issued a request for proposals (RFP) seeking contract bids for enhancing the capacity of the PIT System. CST submitted a proposal, but ECC chose not to award the contract and performed the work on the PIT System itself.

Plaintiffs claim that: (i) ECC performed this enhancement, a modification that resulted in an operation of the PIT System at an unlicensed rate; [FN3] (ii) defendant allowed ECC to perform this work without a license; and (iii) defendant has continued to use plaintiffs' patents without license since 1993. Plaintiffs filed their complaint in this court on January 13, 1997, seeking compensation under 28 U.S.C. § 1498 for defendant's infringement of all 25 of the claims contained in the 800 patent. Plaintiffs also seek just compensation for defendant's unlicensed use of plaintiffs' patented technology under the takings clause of the Fifth Amendment.

FN3. The PIT System was sold under a

license to treat 100 gallons per minute and to precipitate out 40 pounds of minerals (specifically copper, a non-ferrous mineral) a day; following modification by ECC, the PIT System was operating at a minimum of 300 gallons per minute and 560 pounds of copper precipitation per day.

Defendant filed its motion for partial summary judgment on April 28, 1999, responding to the claim of infringement by asking this court to declare the 800 patent invalid due to plaintiffs' violation of 35 U.S.C. § 102(b). [FN4] Defendant claims that the PIT System was offered for sale more than one year before May 25, 1993, the critical date upon which the plaintiffs first applied for a patent, and that the PIT System was "ready for patenting" on or prior to this date. In support of this claim, defendant relies, *inter alia*, on deposition testimony given by Mr. Stevenson on March 26, 1998, in litigation between ECC and CST, which it contends suggests that the PIT System was virtually complete in 1990 or 1991. [FN5]

FN4. Section 102(b) of Title 35 provides, in pertinent part, that "[a] person shall be entitled to a patent unless--

(b) the invention was ... in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States."

The measuring date for purposes of applying this subsection--the date of the patent application from which the one-year period is measured--is often referred to as the "critical date."

FN5. This deposition testimony reads, in pertinent part:

Q: When did the metals removal process that you incorporated into the Indiana, Pennsylvania, unit become complete or finalized?

A: I think it became finalized sometime in '90, '91, in that area.

Q: Why didn't you apply for a patent at that time?

A: Because there were other developments and enhancements I wanted to add before I went for the patent application.

Q: Between '90 or '91 and the time you applied for the patent, May 25, 1993, what other developments or enhancements to the metals removal process did you include?

A: Primarily control and the need to eliminate a large piece of equipment we don't need any more.

In opposing this motion, plaintiffs argue that the subject of the 800 patent was not the PIT System, *per se*, but rather a method hinging on the particular mix and ordering of flocculating agents used within the PIT System. They contend that while this polymer chemistry had been reduced to practice for ferrous metals before the critical date, as evidenced by its use at the Indiana, Pennsylvania, \*516 site, it was not developed for copper-laden pollutant streams, such as that present in Summitville, which involved both ferrous and nonferrous minerals, until several months after the critical date. As such, they contend there was no violation of the "on-sale bar" as to the method underlying the PIT System and incorporated into the 800 patent.

## II. Discussion

Summary judgment is as viable in a patent case as in any other. *See Becton Dickinson & Co. v. C.R. Bard, Inc.*, 922 F.2d 792, 795 (Fed.Cir.1990). Summary judgment is appropriate when there is no genuine dispute as to any material fact and the moving party is entitled to judgment as a matter of law. *See* RCFC 56; *Anderson v. Liberty Lobby, Inc.*, 477 U.S. 242, 247-48, 106 S.Ct. 2505, 91 L.Ed.2d 202 (1986). Disputes over facts that are not outcome-determinative under the governing law will not preclude the entry of summary judgment. *See Anderson*, 477 U.S. at 248, 106 S.Ct. 2505. However, summary judgment will not be granted if "the dispute about a material fact is 'genuine,' that is, if the evidence is such that a reasonable [trier of fact] could return a verdict for the nonmoving party." *Id.* *See also Matsushita Elec. Indus. Co., Ltd. v. Zenith Radio Corp.*, 475 U.S. 574, 587, 106 S.Ct. 1348, 89 L.Ed.2d 538 (1986); *California ex rel. Dept. of Transp. v. United States*, 27 Fed.Cl. 130, 135 (1992), *aff'd*, 11 F.3d 1071, 1993 WL 410284 (Fed.Cir.1993).

When reaching a summary judgment determination, a judge's function is not to weigh the evidence, but to determine whether there is a genuine issue for trial. *See Anderson*, 477 U.S. at 249, 106 S.Ct. 2505. *See also Agosto v. INS*, 436 U.S. 748, 756, 98 S.Ct. 2081, 56 L.Ed.2d 677 (1978) ("[A] [trial] court generally cannot grant summary judgment based on its assessment of the credibility of the evidence presented"). The judge must determine whether the evidence presents a disagreement sufficient to require submission to fact finding, or whether it is so one-sided that one party must prevail as a matter of law. *See Anderson*, 477 U.S. at 250-52, 106 S.Ct. 2505. In doing this, all facts must be construed in a light most favorable to the nonmoving party and all

inferences drawn from the evidence must be viewed in the light most favorable to the party opposing the motion. *See Matsushita*, 475 U.S. at 586-87, 106 S.Ct. 1348 (citing *United States v. Diebold*, 369 U.S. 654, 655, 82 S.Ct. 993, 8 L.Ed.2d 176 (1962)).

Under 35 U.S.C. § 282 (1994 & Supp. III 1997), a patent is presumed valid and one challenging its validity bears the burden of proving invalidity by clear and convincing evidence. *See Mas-Hamilton Group v. LaGard, Inc.*, 156 F.3d 1206, 1216 (Fed.Cir.1998); *Innovative Scuba Concepts, Inc. v. Federated Indus. Inc.*, 26 F.3d 1112, 1115 (Fed.Cir.1994). An issued patent is invalid if "the invention was ... on sale in this country, more than one year prior to the date of the application for patent in the United States, ..." 35 U.S.C. § 102(b). The sale of the invention, which case law makes clear includes an offer for sale, [FN6] must be for commercial gain and not merely for experimental use. The ultimate determination whether an invention was on sale within the meaning of section 102(b) is a question of law, based on the underlying facts. *See Tec Air v. Denso Manufacturing Michigan, Inc.*, 192 F.3d 1353, 1357 (Fed.Cir.1999); *Weatherchem Corp. v. J.L. Clark, Inc.*, 163 F.3d 1326, 1332 (Fed.Cir.1998).

FN6. *See, e.g., D.L. Auld Co. v. Chroma Graphics Corp.*, 714 F.2d 1144, 1150 (Fed.Cir.1983).

In *Bonito Boats, Inc. v. Thunder Craft Boats, Inc.*, 489 U.S. 141, 151-52, 109 S.Ct. 971, 103 L.Ed.2d 118 (1989), the Supreme Court explained that the "federal patent system ... embodies a carefully crafted bargain for encouraging the creation and disclosure of new, useful, and nonobvious advances in technology and design in return for the exclusive right to practice the invention for a period of years." "In consideration of its disclosure and the consequent benefit to the community," the Supreme Court further stated, "the patent is granted" and the investor receives an exclusive monopoly for a limited period of time. 489 U.S. at 151, 109 S.Ct. 971. In this statutory context, section 102 "serves as a limiting provision ... confining the \*517 duration of the monopoly to the statutory term." *Pfaff v. Wells Electronics, Inc.*, 525 U.S. 55, 119 S.Ct. 304, 310, 142 L.Ed.2d 261 (1998). *See also King Instrument Corp. v. Otari Corp.*, 767 F.2d 853, 860 (Fed.Cir.1985).

In *Pfaff*, the Supreme Court enunciated the standard for applying the "on sale bar." Wayne K. Pfaff designed computer chip sockets. *See* 119 S.Ct. at 307. In 1980, Texas Instruments contacted Pfaff and asked him to design a special computer chip socket

for the company. *Id.* Pfaff prepared detailed engineering drawings that described the design, the dimensions and the material to be used in the making of the socket. *Id.* Before the critical date, Pfaff showed a sketch of his concept to Texas Instruments. *Id.* Subsequently, but prior to the critical date, Texas Instruments forwarded a written order to Pfaff for the purchase of 30,100 sockets. *Id.* Although the invention was not reduced to practice and the order was not filled until after the critical date, the Supreme Court concluded that the "invention had been on sale for more than one year in this country before [Pfaff] filed his patent application." 119 S.Ct. at 312.

In reaching its decision, the Supreme Court, recognizing the desirability of developing a "bright-line rule," held that two conditions were required before the on sale bar applied. "First, the product must be the subject of a commercial offer for sale.... Second, the invention must be ready for patenting." 119 S.Ct. at 311-12. The Court held that to demonstrate that an invention is ready for patenting a party could prove, *inter alia*, either that the invention was reduced to practice before the critical date or, "that prior to the critical date the inventor had prepared drawings or other descriptions of the invention that were sufficiently specific to enable a person skilled in the art to practice the invention." *Id.* at 312. [FN7] Because Pfaff's drawings allowed the manufacturer to produce the socket, and the sockets manufactured contained all the elements of the invention, the Supreme Court held that the invention was ready for patenting. *Id.* [FN8]

FN7. *See generally, Isabelle R. McAndrews, "The On-Sale Bar After Pfaff v. Wells Electronics: Toward a Bright-Line Rule,"* 81 J. Pat. & Trademark Off. Soc'y 155 (1999).

FN8. The Federal Circuit has repeatedly applied *Pfaff* in its recent cases, noting that the decision supplants the Federal Circuit's prior "totality of the circumstances" analysis of the "on sale doctrine." *See Brasseler, U.S.A. I, L.P. v. Stryker Sales Corp.*, 182 F.3d 888, 889- 90 (Fed.Cir.1999); *Weatherchem Corp.*, 163 F.3d at 1333.

[1] Applying the *Pfaff* test to the facts of this case requires the defendant to establish the absence of a dispute of material fact in respect to the two elements of the "on sale" defense, namely, (i) that there was a definite commercial sale or offer for sale of the invention claimed in the 800 patent more than one year before the plaintiffs filed their patent application;

and (ii) that at the time of that sale or offer for sale, the invention was ready for patenting. While the defendant has met its burden as to the first prong of this test, the court concludes that material questions of fact exist as to the second prong of the *Pfaff* test, i.e., whether the invention was ready for patenting.

As to the first prong of the *Pfaff* test, it appears uncontested that the PIT System--encompassing both an apparatus and chemical process--was on sale prior to the critical date, May 25, 1992. In this regard, defendant correctly points out that in advance of the critical date, plaintiffs had made an offer to Summitville, that offer had been accepted, a purchase order had been submitted by Summitville, and Summitville had made its first lease payments to Vision Financial, a leasing agent facilitating the transaction between CST and Summitville. The existence of these events is clear and convincing proof that the invention in question was subject to a commercial offer prior to the critical date. *See Pfaff*, 119 S.Ct. at 311 (acceptance of purchase order makes clear that commercial offer made); *Weatherchem*, 163 F.3d at 1333 (signed purchase agreement evidence of commercial offer). *See also Evans Cooling Systems, Inc. v. General Motors Corp.*, 125 F.3d 1448, 1451 (Fed.Cir.1997), cert. denied, 522 U.S. 1115, 118 S.Ct. 1050, 140 L.Ed.2d 113 (1998) (discussing similar evidence of commercial offer).

\*518 Plaintiffs attempt to counter these facts by alleging that the sale to Summitville was an experimental use. To be sure, a sale of the flocculation method for experimentation rather than profit would not constitute a commercial sale for purposes of applying the first prong of the *Pfaff* test. *See Pfaff*, 119 S.Ct. at 311; *Zacharin v. United States*, 43 Fed.Cl. 185, 192 (1999). However, the Federal Circuit requires solid proof that the sale of an invention be "substantially for purposes of experiment," basing the existence of such a defense on "the objective evidence of experimentation, including the number of prototypes and duration of tests conducted, whether test records and progress reports were kept, the existence of a secrecy agreement, whether the investor received compensation for use of the invention, and the extent to which the inventor controlled the testing." *Zacharin*, 43 Fed.Cl. at 192. *See also LaBounty Mfg., Inc. v. United States, Int'l Trade Comm'n*, 958 F.2d 1066, 1071-74 (Fed.Cir.1992). Here, plaintiffs have produced no evidence that such records and experimental protocols existed with respect to the Summitville sale.

[2] The fact that the sale to Summitville was for commercial purposes, however, does not resolve the

second prong of the *Pfaff* test, i.e., whether the invention was ready for patenting. [FN9] Indeed, material questions of fact exist as to this prong of the analysis. In particular, further evidence is needed to determine whether the product on sale before the critical date was the "invention later claimed" [FN10]--that is whether a comparison of the claims in the 800 patent to the PIT System in use at Indiana, Pennsylvania in 1991 and sold to Summitville in 1992 reveals that essentially the same invention or inventions were involved.

FN9. At first blush, it might seem odd to conclude that the sale to Summitville was not for experimental purposes, but that it is unclear whether the invention was ready for patenting. But, the two prongs of the *Pfaff* test are not focused on the same phenomenon. The first prong looks, *inter alia*, to whether a prior sale was for profit, as opposed to experimental purposes, while the second prong focuses on the state of the invention itself. Given this, it appears conceivable that an inventor could sell or lease an invention for profit prior to the time the invention was fully developed. Indeed, this theoretical possibility must exist lest the two prongs of the *Pfaff* test converge into one--whether the invention was for sale commercially. Notably, prior to *Pfaff*, some decisions had employed this single-prong approach in deciding whether the "on sale bar" applied. *See, e.g., Paragon Podiatry Laboratory, Inc. v. KLM Laboratories, Inc.*, 984 F.2d 1182, 1187 n. 5 (Fed.Cir.1993)("[T]he thrust of the on-sale inquiry is whether the inventor thought he had a product which could be and was offered to customers, not whether he could prevail under the technicalities of reduction to practice"); *UMC Electronics Co. v. United States*, 816 F.2d 647, 657 (Fed.Cir.1987)("[T]he development of the subject invention was far beyond mere conception ... and had been sufficiently tested to demonstrate to the satisfaction of the inventor that the invention as ultimately claimed would work for its intended purpose."). However, in *Pfaff*, the Supreme Court explicitly rejected this approach, noting that "the possibility of additional development after the offer for sale in these circumstances counsels against adoption of [this] rule." *Pfaff*, 119 S.Ct. at 312 n. 14.

FN10. *See Scaltech, Inc. v. Retec/Tetra*,

While defendant argues that the record is clear in this regard, the court does not agree, particularly when, as it must, it views the evidence and draws factual inferences favorably to the plaintiff, and especially in light of the clear and convincing evidence standard imposed on the defendant. Thus, for example, it is unclear from the record how critical the polymer chemistry (i.e., not only the polymers chosen, but when and how in the process those polymers were introduced) was to the success of the patented method and, if critical, when that chemistry was patentable. [FN11] Moreover, a material dispute exists as to whether the allegedly essential polymer chemistry was developed prior to the critical date, as defendant claims, or on or around August 5, 1992, as plaintiffs claim. Relatedly, \*519 questions exist as to whether the work done by Mr. Stevenson in the weeks leading up to August 5, 1992, was experimentation going to the core of the patent or merely fine tuning. *See Weatherchem*, 163 F.3d at 1334 (invention can be ready for patenting, even though an inventor continues to "fine-tune features not claimed in the patent"). Along these lines, further evidence is also needed to determine to what extent the chemistry required to flocculate non-ferrous metals was different from that necessary to flocculate ferrous metals--the latter process having been employed successfully by plaintiffs in 1991 at the Indiana, Pennsylvania site. Finally, also relevant to the second prong of the *Pfaff* analysis is information concerning the nature of the guarantee made by the plaintiffs to Summitville--that is, was it a typical money-back guarantee or a guarantee that highlighted the fact that the polymer chemistry had not yet been worked out. *See, e.g., LaBounty*, 958 F.2d at 1074 (noting that a "money-back guarantee is a typical commercial sales provision and does not establish an experimental relationship between the parties.").

FN11. The Patent and Trade Office's file regarding the 800 Patent, which was jointly provided by the parties in supplementation of the record, adds an additional layer of mystery to this inquiry. From that file, it appears that the patent examiner initially was disinclined to grant the requested patent because he felt that the underlying chemistry was obvious. However, the patent examiner subsequently concluded that some aspect of the plaintiffs' use of polymers--perhaps the order in which aeration, neutralization, agitation and flocculent addition occurred--went beyond prior art and he thus granted the patent.

The plaintiffs thus have demonstrated a number of issues of material fact still in dispute that prevent this court, at this juncture, from finding in favor of either party.

### III. Conclusion

In sum, based on the foregoing, the court concludes that the allegations concerning the validity of the 800 patent are not resolvable without a trial. Accordingly, defendant's motion for partial summary judgment is DENIED.

END OF DOCUMENT

COPY

May 15, 1992

Summitville Consolidated Mining Co., Inc.  
P.O. Box 2G  
Del Norte, Colorado, USA 811362

Attention Mr. Bill Williams

Subject: Galactic/Summitville  
C.S.T. Mobile Caustic Treatment and  
Dewatering Systems

EXHIBIT

30

Dear Bill,

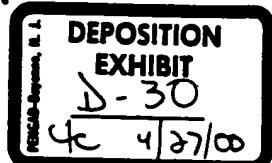
I hope the following information along with the drawings sent to you by Federal Express, 5/14/92, is the information you require.

CST units are adaptable to alternate energy, oxidation and chemical inputs to treat and dewater most waste water discharges of acidic or alkaline nature.

CST neutralization units optimize chemical use and eliminates continued operator labor input. At present we have units in the field capable of treating raw water from 150 GPM to 2500 GPM.

CST units servo automatically for changes in flow, temperature, acidity, alkalinity, heavy metals and neutralizer strength. All controls and sensors are interfaced with a programmable controller. An aerator/mixer oxidizes the metals and mixes the continuous process. All components are combined in a compact patented method to facilitate an instantaneous reaction and optimize chemical consumption.

CST units are designed and built to an owner's particular site and/or water quality requirements.



D004002

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INNOVATIVE ENVIRONMENTAL TECHNOLOGIES

Post Office Box 931 • McMurray, Pennsylvania 15317 • (412) 942-0679 • FAX (412) 942-0436

# CHEMICAL SEPARATION TECHNOLOGY, INC.

Summitville Consolidated Mining Co., Inc.  
Page 2  
May 15, 1992

The design criteria for your Reynolds ADIT site is as follows:

## Water Quality Worst Case

Flow	100 GPM
pH	2.98
Acidity	2200 Mg/l
Iron Fe++	460 Mg/l
Copper	190 Mg/l
Managanese	26 Mg/l
Zinc	34 Mg/l

Utilization of 50% NaOH solution at 100 GPM raw water and 2200 Mg/l acidity, will be approximately 14 gallons per hour respectively in the worst case scenario.

## Operational Sequence:

Raw water shall be pumped from a holding pond to the CST reactor at 100 GPM.

Sodium Hydroxide and oxygen will then be added and mixed with the raw water to boost the pH instantly to 8.5 (other pH values can be programmed).

Neutralized/oxidized effluent is then discharged from the reactor to the clarifier/settling tank.

Supernate from the clarifier/settling tank is then discharged to the polishing tank.

Clean supernate is then discharged from the polishing tank to the stream.

When sludge buildup in the clarifier/settling tank is to within 12" from the supernate discharge port the treatment system will be shut down and dewatering of the sludge will begin.

Sludge will be pumped at approximately 2% solids to the rotary thickener flocculation tank where polymers will be

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004003

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# CHEMICAL SEPARATION TECHNOLOGY, INC.

Summitville Consolidated Mining Co., Inc.  
Page 3  
May 15, 1992

added. The sludge continues thru the rotary thickener to the discharge hopper where it will be pumped or conveyed to a dump truck for disposal.

The system components are as follows:

- 1) One (1) truck mounted treatment system
- 2) One (1) 9,000 gallon skid mounted storage tank
- 3) Emergency shower
- 4) Two (2) 21,000 gallon mobile clarifiers
- 5) One (1) 100 GPM rotary thickener complete
- 6) Polymer dosing units
- 7) One (1) loading conveyer

Summitville shall supply and maintain the following:

- 1) An accessible haul road to the building
- 2) One (1) 100 AMP 480 VAC 3 phase 60 HZ power source to within 10 feet of the MCTS
- 3) Telephone line
- 4) Potable water line
- 5) Raw water collection pond
- 6) Raw water pumping
- 7) Piping or miscellaneous hoses and fittings from the raw water collection pond
- 8) Chemicals (caustic and emulsion polymers)
- 9) Plant service personnel - CST to train
- 10) Sludge hauling and disposal

0004004

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Post Office Box 931 • McMurray, Pennsylvania 15317 • (412) 942-0679 • FAX (412) 942-0436

**CHEMICAL SEPARATION TECHNOLOGY, INC.**

Summitville Consolidated Mining Co., Inc.  
Page 4  
May 15, 1992

Please call me if you need any additional information.

Very truly yours,

Chemical Separation Technology, Inc.



S.M. Stevenson,  
President

SMS/tm

201409

D004005

**INNOVATIVE ENVIRONMENTAL TECHNOLOGIES**

Post Office Box 931 • McMurray, Pennsylvania 15317 • (412) 942-0679 • FAX (412) 942-0436

**CHEMICAL SEPARATION TECHNOLOGY, INC.**

May 15, 1992

Summitville Consolidated Mining Co., Inc.  
P.O. Box 2G  
Del Norte, Colorado, USA 81132

Attention Mr. Bill Williams

Subject: Metals Removal Reynolds ADIT

Dear Bill,

Per our telephone conversation yesterday, you inquired about additional metal recovery at the Reynolds ADIT Site as follows:

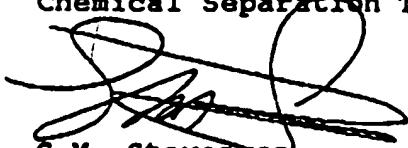
Mercury	<.0001 Mg/L
Lead	.37 Mg/L
Cadmium	.25 Mg/L

As I explained, the above metals will experience some co-precipitation with Fe and Cu. However, the extent of the co-precipitation will have to be determined once the system is on site and all chemical additions and pH adjustments are operating properly. The first few sets of internal plant analyses should give us the necessary baseline to determine the amount of co-precipitation of these metals.

If you need any additional information for your report, please contact me at your convenience.

Very Truly Yours,

Chemical Separation Technology, Inc.

  
S.M. Stevenson

SMS/tm

201411  
0004007

INNOVATIVE ENVIRONMENTAL TECHNOLOGIES

Post Office Box 931 • McMurray, Pennsylvania 15317 • (412) 942-0670 • FAX (412) 942-0436

**CHEMICAL SEPARATION TECHNOLOGY, INC.**

**CST**

**Client List**

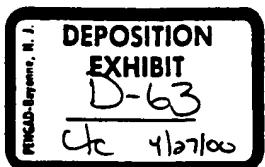
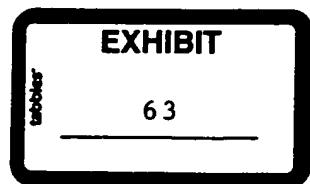
Waste Water Treatment Neutralization Facility Turnkey Project and Services	Aloe Coal Midway, PA 1986
Waste Water Treatment Neutralization Facility Turnkey Project and Services	Mincorp Somerset, PA 1986
Waste Water Treatment Neutralization Facility Turnkey Project and Services	Penelec-Keystone Station Shelocta, PA 1987
Waste Water Treatment Neutralization Facility Feasibility Study	British Petroleum, Inc. Cleveland, OH 1988
Waste Water Treatment Neutralization Facility Feasibility Study	Consolidation Coal Co. Pittsburgh, PA 1988
Waste Water Treatment Neutralization Facility Feasibility Study	Old Ben Coal Cleveland, OH 1988
Waste Water Treatment Neutralization Facility Feasibility Study	Homestake Mining California 1988
Waste Water Treatment Neutralization Facility Turnkey Project and Services	Old Ben Coal J. R. Plant, Benton, IL 1989
Sludge Dewatering Facility Turnkey Project and Services	Penelec-Keystone Station Shelocta, PA 1989
Waste Water Treatment Neutralization Facility Turnkey Project and Services	LTV Steel Company Meadowlands, PA 1991

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0004008

# **CHEMICAL SEPARATION TECHNOLOGY, INC.**

## **Business Plan and Financial Forecast**

**July, 1991**



**CST 0121**

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## EXECUTIVE SUMMARY

Chemical Separation Technology, Incorporated (CST) is a water treatment service company with offices located in Vancouver, Washington, and Pittsburgh, Pennsylvania. CST provides services focused towards utility companies and the metals and coal mining industries. The Company installs its high-tech acid neutralization treatment units on client's sites to process acidic discharge contaminated with metals in solution, producing an effluent which meets EPA discharge standards. The CST mission statement is to resolve environmental discharge problems for clients through the delivery of price competitive treatment services which will allow them to achieve regulatory compliance and environmental recognition.

CST's core business focus is to own and operate as a turnkey provider of water treatment services, addressing each client's particular environmental discharge problems. We do not intend to market our patented equipment for resale as a manufacturer. CST operations are supported by principals with broad experience in organization, planning, management, operations, engineering, technological and financial matters and also possess strong expertise and contact networks in the utility and mining industries.

CST is a patented process (United States Patent Number 4,749,497) which provides a unique method for treatment of acidic and metals-contaminated leachates. The patented CST equipment is comprised of a cylindrical reaction vessel with internal baffles, computer-controlled pH monitoring and recording equipment, an aeration motor, piping and chemical storage tanks. The equipment is easily scaled to handle volume flow rates ranging from as small as ten gallons per minute to major flows of 10,000 gallons per minute. For flow rates from ten to 500 gallons per minute the CST reactor and ancillary equipment is portable, being either skid-mounted or installed in van-type trailers. The capital cost for a CST unit is significantly less than alternative lime base treatment systems.

The CST process combines sodium hydroxide and oxygen with the leachate in a patented reactor to neutralize acid and precipitate metals. Air is continuously drawn into the reactor in the form of very fine bubbles and mixed simultaneously with concentrated sodium hydroxide to create localized pockets of high oxygen concentration and alkalinity. Use of state-of-the-art computerized process control instrumentation holds the pH at a constant level allowing point precipitation of metals in solution. The net effect of this design is an acceleration in the oxidation and precipitation rates which are considerably higher than those achieved by conventional mechanical agitated, lime-based processes. Sludge thickening, dewatering, clarification and filtration steps, reduce solid wastes to minimal volume in order to recover the precipitated metals for efficient disposal or reuse.

Competitive processes focus on the addition of lime or lime-sodium carbonate to increase the pH. The resultant slurry is mixed to promote neutralization and oxidation. These processes require large settling basins or ponds to permit the mixing and slow precipitation to occur. Control of pH with lime depends on the extent of solid-liquid mixing and the reactivity of the lime and provide only a high pH range. Generally, such treatment facilities are very capital intensive, utilizing complex mixing, lime slaking, and other labor intensive equipment. Additionally, while solids from lime processes include the precipitated metals, they also produce large quantities of unreacted lime which creates extensive material handling requirements.

The primary markets for CST systems are utility power generation plants, coal mines and metals mines. Acidic and metals-bearing leachates from coal-fired utility power plant operations and flyash deposits, as well as discharge from coal mines and metals mines, severely degrade streams, rivers or lakes unless remedial actions are implemented. An increasing public concern for the quality of water released to the environment and compliance with the Clean Water Act, mandates corrective actions be identified and implemented. The increasing threat of civil and criminal penalties for non-compliance with the EPA Standards has accelerated the demand for technological solutions to complex discharge problems.

The number of opportunities to provide treatment services for utilities, coal mines and metals mines is enormous. There are over 800 coal-fired power plants which have operations conducive to the application of CST equipment. Changes in the Clean Air Act will require installation of scrubbers at a majority of these generation facilities over the next decade. Process waters associated with operation of the scrubbers will require treatment under new EPA Standards before being discharged. In the coal mining area there are approximately 4,900 active mines which require treatment of some magnitude. Additionally, there are thousands of abandoned coal mine sites in all parts of the United States, as well as Eastern Europe, where significant problems exist. The metals mining industry, particularly in the Western United States, operated virtually in the absence of environmental regulations for a period of over a hundred years. There are literally hundreds of active and abandoned mine sites which, with the increase in environmental sensitivity, will be required to address their discharge problems or face fines and possible interruption of their operations. New sites seeking permitting will be required to meet the most stringent new standards.

CST currently has two sites in commercial operation. One is at the Pennsylvania Electric Company Keystone Power Generation Station. The utility plant is an 1800 MW coal-fired generation unit located near Indiana, Pennsylvania. The second is for LTV Steel Company (LTV) at a coal refuse disposal dump near the site of an abandoned deep coal mine in Western Pennsylvania. The kinds of results routinely achieved at the power plant operation produces effluent acceptable for industrial use and/or discharge to the local watershed. In excess of 99.9% of the iron and 94% of the manganese are precipitated in less than 10 minutes at a nominal pH of 7-8.

Liquid-solid separation processes using clarification and belt filtration recover approximately 15,000 pounds of solids daily. The facility routinely accepts and treats higher level excursions of acidity and metals in solution up to 6,000 mg/l and 7,000 mg/l respectfully. At the LTV site, their current Pennsylvania DEQ permit allows the leachate to be neutralized and discharged into a bore hole to an abandoned mine shaft where the metals precipitate out of solution. More recent regulatory permitting would require a liquid-solid separation processes to recover the precipitated metals for shipment to an approved disposal site.

As of this time CST has not implemented its formal marketing program. Corporate marketing objectives focus on rapid penetration of the market to obtain market share and reinvestment in continued Research and Development to remain on the leading edge of treatment technology. We believe the window to achieve near-term market share in this emerging marketplace is very dynamic. As time passes the market growth will levelize as new competitors launch intense efforts to develop competing technologies. This is particularly true as it relates to Superfund Sites where development of alternative technologies is actively supported by very large companies and financed with Federal Government funding. Achieving rapid market penetration goals presents an ambitious implementation objective but presents the most viable opportunity for sustaining long-term growth.

In 1990 CST produced net income of \$64,523 on revenues of \$414,510. For the six month period ending June 30, 1991, CST has net income of \$9,733 on revenues of \$189,665. The substantial decline in percentage of net income for the most recent period is the result of expenditures made in expanded engineering and marketing activities.

CST has compiled a ten year financial forecast, including forecasted balance sheets, statements of earnings, retained earnings and cash flows for the period ending in the year 2000. These forecasts were developed in accordance with standards established by the American Institute of Certified Public Accountants. Appropriate market and sales assumptions are described in detail in the forecast. Based on conservative projections it appears the Company could achieve annual revenues and net income of \$124 million and \$12 million by 1995 and \$523 million and \$53 million respectively by the year 2000. It is essential to note that as project unit financing is amortized, cash flows generation improves significantly. Another fundamental point which impacts net income and requires mention, is the Company's focus on reinvestment of cash flow in Research and Development. Details supporting these financial forecast results are contained in the body of this document.

CST's immediate goal is to focus on market development and accelerated financial growth. As an emerging company it recognizes the magnitude of the impending challenge to attain this goal. To achieve the accelerated expectations, there is compelling evidence of the need to align its operations with a cooperative entity possessing a substantial financial and synergistic infrastructure. While CST remains

flexible in analyzing various approaches, we continue to believe the most solid framework for implementing mutually beneficial objectives, is by offering an equity position to an entity which has compatible characteristics. These positive attributes would include: visionary and aggressive attitude surrounding business approach; skill sets which compliment the elements where CST requires enhancement; and the financial strength to make a substantial capital investment. Business experience indicates the probability for success improves dramatically when all participants have a vested equity interest and are committed to the future success of the company.

We believe CST has an extremely bright future in the environmental water treatment business. The initial response to our technology and business approach has been exceedingly positive. Our primary objective is to establish a financial and operational infrastructure relationship which is commensurate with our aggressive strategic plan. This will provide the Company the ability to quickly become a proactive force in the environmental water treatment marketplace.

## CONFIDENTIALITY AGREEMENT

Copy No. \_\_\_\_\_ provided to \_\_\_\_\_.

**Confidentiality of Information** - This prospectus document is for your confidential use only. It is being submitted to prospective investor or shareholders of Chemical Separation Technology, Inc., solely for your confidential use, with the express understanding that, without the prior written permission of Chemical Separation Technology, Inc., you will not release this document or discuss the information contained herein or make reproductions of or use this document for any purpose other than evaluating the potential opportunity associated with becoming an investor or shareholder in the Company.

A prospective investor or shareholder, by accepting delivery of this document, agrees to promptly return to Chemical Separation Technology, Inc., this document and any other documents or information furnished if the prospective investor or shareholder does not elect to become a contributing investor or shareholder in Chemical Separation Technology, Inc.

The confidentiality agreement on the following pages is intended to be a mutually beneficial and protective instrument. This agreement will bind both signatory parties to keep confidential the sensitive technical and business information of the other. The purpose of this agreement is to allow the free exchange of information which could be used to conclude a mutually cooperative business relationship.

## CONFIDENTIALITY AGREEMENT

THIS AGREEMENT is entered into this \_\_\_\_\_ day of June, 1991, by and between CHEMICAL SEPARATION TECHNOLOGY, INC. (hereinafter "CST"), having an office at 9817 N.E. 54th Street, Suite 101, Vancouver, Washington 98662, and \_\_\_\_\_ (hereinafter "\_\_\_\_\_"), having an office at \_\_\_\_\_.

WHEREAS, CST, and \_\_\_\_\_ may desire to enter into negotiations for a possible joint cooperative relationship for the utilization of CST technology and to negotiate various business ventures between CST and \_\_\_\_\_; and

WHEREAS, during these negotiations, each party will be exposed to proprietary information, including technology and business plans of the other party; and

WHEREAS, each party desires to enter into this agreement for the protection of its inventions, proprietary information and business plans from disclosure or use by the other party;

NOW, THEREFORE, in consideration of the premises and for the purposes of facilitating the negotiations, CST and \_\_\_\_\_, each corporation, employees, agents and assigns, agree as follows:

A. During the term of all negotiations and for a period of five (5) years after the conclusion of negotiations between CST and \_\_\_\_\_ each party shall keep confidential and refrain from using or disclosing to others all technological information, proprietary information, trade secrets and business plans of the other party that is acquired or learned in the course of negotiations between the parties. All written information shall be clearly marked proprietary by the owning party. All confidential conversations shall be described as such by the owning party.

B. This nondisclosure obligation is not intended to prevent either party from making disclosures required by applicable law or by the order of any court having jurisdiction; provided, however, that a party intending to make a disclosure required by law or by a court order shall use its best efforts to provide to the other party reasonable written notice of such intent prior to disclosure and to cooperate with the lawful efforts of the other party to oppose or resist such disclosure.

C. Upon termination of negotiations, and whether or not a joint venture or other agreement is reached by the parties, each party shall return to the other all documents, records, notebooks and other sources containing the other party's Proprietary Information.

D. If a party to this Agreement believes that information claimed to be confidential under this Agreement by the other party is not in fact confidential, it shall notify the other party and, prior to disclosure, shall meet with the other party and discuss the confidential nature of the information.

E. No rights under this Agreement shall be assigned or transferred by either party without the written consent of the other party.

F. No governmental agency or private party, including any subcontractors, is intended to be a third party beneficiary of any rights or obligations under this Agreement.

G. This Agreement shall be construed under the laws of the State of Idaho. Exclusive venue for any claims arising under this Agreement shall be in the appropriate court in Boise, Idaho.

H. Each party acknowledges that disclosure of Proprietary Information would cause irreparable harm to the other party, and agrees that the obligations of this Agreement may be enforced by an injunction or order of specific performance, and that a party harmed by such disclosure shall be entitled to damages and all other remedies allowed by law.

I. The terms of this Confidentiality Agreement shall be considered to be a part of each and every agreement that may be entered into between the parties unless expressly waived by the parties.

CHEMICAL SEPARATION  
TECHNOLOGY, INC.

By \_\_\_\_\_

By \_\_\_\_\_

Its \_\_\_\_\_

Its \_\_\_\_\_

STATE OF \_\_\_\_\_ ) ss.

County of \_\_\_\_\_ )

On this \_\_\_\_\_ day of \_\_\_\_\_, 19 \_\_\_\_\_, before me, the undersigned, a Notary Public in and for the State of \_\_\_\_\_, duly commissioned and sworn, personally appeared \_\_\_\_\_, known to me to be the \_\_\_\_\_ of CHEMICAL SEPARATION TECHNOLOGY, INC., the corporation that executed the foregoing instrument, and acknowledged the said instrument to be the free and voluntary act and deed of said corporation, for the uses and purposes therein mentioned, and on oath state that he is authorized to execute the said instrument and that the seal affixed is the corporate seal of said corporation.

WITNESS my hand and official seal hereto affixed the day and year in this certificate above written.

Notary Public in and for  
the State of: \_\_\_\_\_  
Residing at: \_\_\_\_\_  
My Commission expires: \_\_\_\_\_

STATE OF \_\_\_\_\_, ) ss.  
County of \_\_\_\_\_ )

On this \_\_\_\_\_ day of \_\_\_\_\_, 19\_\_\_\_\_, before me, the undersigned, a  
Notary Public in and for the State of \_\_\_\_\_, duly commissioned and sworn,  
personally appeared \_\_\_\_\_, known to me to be the  
\_\_\_\_\_, of \_\_\_\_\_, the corporation that executed  
the foregoing instrument, and acknowledged the said instrument to be the free and voluntary act and deed  
of said corporation, for the uses and purposes therein mentioned, and on oath state that he is authorized  
to execute the said instrument and that the seal affixed is the corporate seal of said corporation.

WITNESS my hand and official seal hereto affixed the day and year in this certificate above  
written.

\_\_\_\_\_  
Notary Public in and for  
the State of: \_\_\_\_\_  
Residing at: \_\_\_\_\_  
My Commission expires: \_\_\_\_\_

## INTRODUCTION

### What is CST

Chemical Separation Technology, Incorporated (CST) is an Idaho Corporation with offices located in Vancouver, Washington, and Pittsburgh, Pennsylvania. CST provides high-tech acid neutralization and waste water treatment services to utility companies and the metals and coal mining industry. The Company installs its treatment units on clients sites to process acidic discharge contaminated with metals suspended in solution. The patented treatment units feature computer controlled caustic neutralization in a specially designed reactor vessel. The portable units are easily moved which is especially attractive for remote discharge and seepage locations. Larger units are installed in either temporary or permanent facilities. The process treatment produces an effluent which meets EPA discharge standards. Site hydrologic characterization, waste water testing and process design engineering services are also offered by the company.

### Mission Statement and Goals

The CST mission is to resolve environmental discharge problems for clients by providing price competitive treatment services which will allow them to achieve regulatory compliance and environmental recognition.

To implement the Company's Mission Statement our operative goals are as follows:

- o Provide safe, efficient and cost effective services which fully satisfies customer requirements
- o Focus near term efforts on rapid market penetration in the utility, coal mining and metals mining industries which will gain a significant market share in this emerging marketplace.
- o Position the Company to achieve continued growth through research and development into new market applications for the CST process.
- o Execute business activities in a profitable manner which will promote the financial health of the Company, provide a stable work environment for employees and maximize return on investment for shareholders.
- o Become a recognized environmental leader and respected corporate citizen through the development of "Innovative Environmental Technologies".

## Corporate Structure

Chemical Separation Technology, Inc. (CST) is an Idaho Corporation partly owned by *Group West* Corporation of Vancouver, Washington and POWER Engineers of Hailey, Idaho. Additionally, there are five individual shareholders who hold equity positions in the Company. The Company was acquired in December, 1990 from the original founders in Pittsburgh, Pennsylvania. An organizational chart showing the current ownership and responsibilities is presented in FIGURE 1 on the following page.

## Personnel Summaries

CST principals have broad experience in organization, planning, management, operations, engineering and financial matters. Additionally, we have expertise, understanding and contact networks in the mining and utility industries. The following are brief personal summaries of the professionals who are the core strength of CST's business expertise.

- o Steven L. Thorson, President - Mr. Thorson's extensive business management and consulting expertise includes experience in utility companies, government agencies and private industry. His consulting knowledge and experience includes direct applications in the following areas: strategic planning; management audits and analysis; organizational restructuring; reorganization advisory counseling; operations improvement; and financial forecasting, budgeting and analysis. Mr. Thorson has developed and implemented a number of computerized models which analyze and forecast appropriate management methodologies in replacement analysis, budgeting, financing, resource allocation and merger/acquisitions. He was formerly a Senior Management Consultant with the firm of Deloitte & Touche. His business activities and consulting assignments have provided him a broad knowledge and understanding of the utility industry and afforded him the opportunity to achieve a strong personal network within the industry. Mr. Thorson holds a Bachelor of Science Degree in Finance from the University of Utah.
- o Russell P. Wischow, Director - Dr. Wischow has been responsible for the development and construction of utility power generation projects and operation and maintenance of major non-regulated independent power projects. Most recently he was the President and CEO of PG&E Operating Services, a non-regulated subsidiary of Pacific Gas and Electric Company which provided operating and maintenance services to the independent power industry. He was the first Director of the USAEC Division of Nuclear Materials Safeguards and was Director of

research and Development for a spent nuclear fuels reprocessing plant which managed the International Atomic Energy Agency sponsored program to develop international inspection of nuclear fuels reprocessing plants. He is a member of various professional societies, holds several patents and has presented papers at national meetings. Dr. Wischow holds a PhD in Chemistry from Vanderbilt University.

- o S. M. Stevenson, Vice President Operations - Mr. Stevenson has twenty eight years of highly diversified experience in the industrial and commercial sector. He is highly qualified in administrative and project management, design and construction coordination and has strong educational background and experience in all levels of management. He is one of the original founders of CST and was instrumental in development of the technology and the CST patent.
- o John J. LeFever, Project Manager - Mr. LeFever is a minerals engineer with 14 years of experience in mine design, engineering geology, projects evaluation, engineering development, and exploration of precious metals, base metals, oil shale, coal, lignite, and salt. As a project manager, he has experience in project budgeting, control and accounting, policy and personnel, as well as in taking a new project from raw prospect through the phases of financial justification and board approval, mining and geological evaluation, detailed engineering, development, and permit approval. Mr. LeFever's background includes six years of experience in computerized geostatistical analysis, ore reserve estimation, mine planning, and cost estimating of open pit precious metals deposits. Mr. LeFever holds a B.S. degree in Geological Engineering from Montana Tech.

### Description of Process

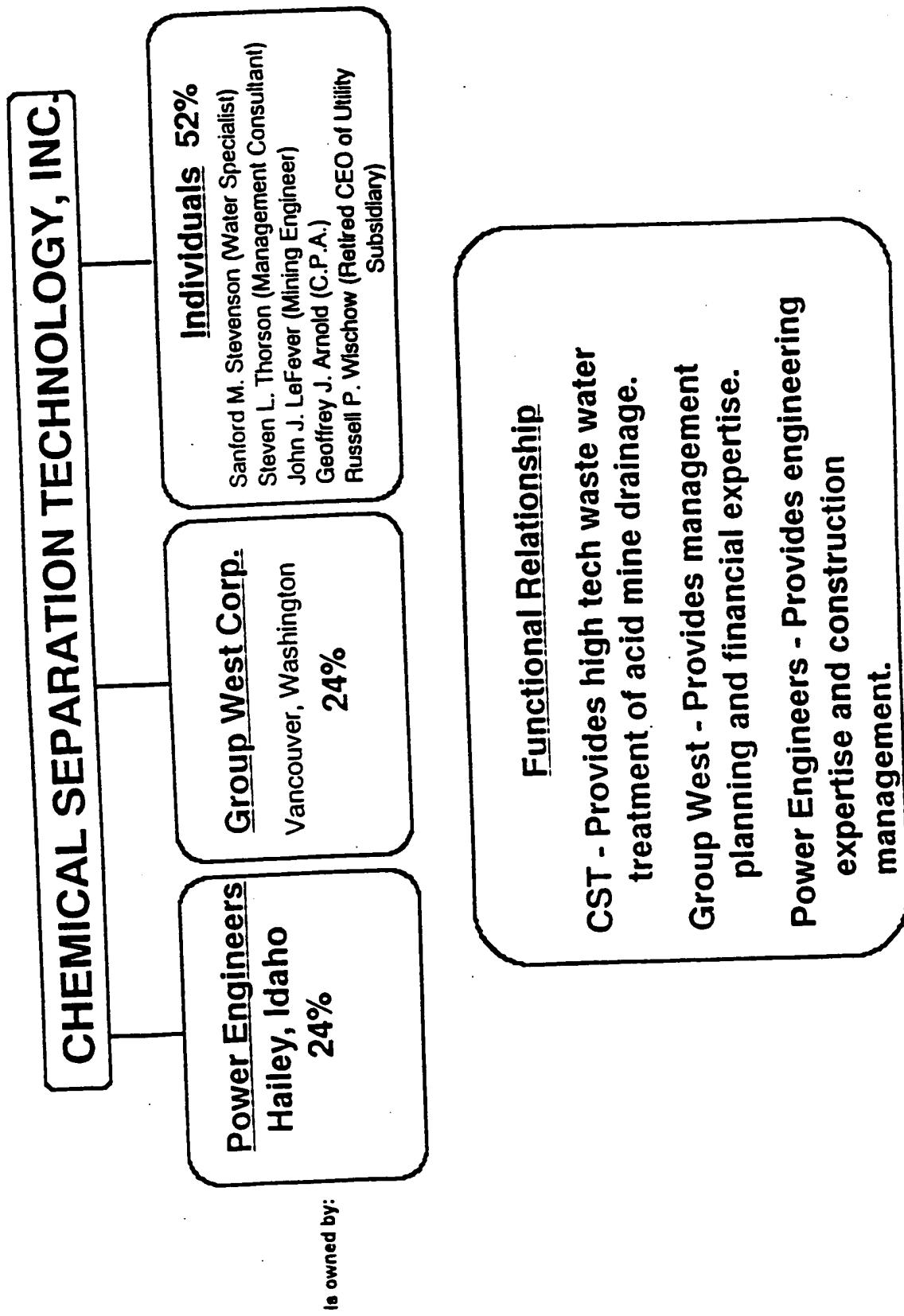
The CST process is currently used to treat leachates in commercial operations at a coal-fired power plant ash disposal site and a coal refuse site in Western Pennsylvania. Sodium hydroxide and oxygen are combined with the leachate in a patented reactor to precipitate metals and neutralize acid. Liquid-solid separation processes are included at the power plant site to recover the precipitated metals for disposal. The CST process differs from other waste water treatment systems in that air, which is continuously drawn into the reactor in the form of very fine bubbles, and concentrated sodium hydroxide, are mixed simultaneously to create localized pockets of high oxygen concentration and alkalinity. The net effect of this design is an acceleration in the oxidation and precipitation rates which are considerably higher than those achieved by conventional mechanical agitated, lime-based processes. Through the use of state-of-the-art computerized process control instrumentation, the pH can be held at a constant

level allowing point precipitation of metals in solution. Reactants other than sodium hydroxide and oxygen, such as soda ash, permanganate, hypochlorite, peroxide and ozone can be incorporated in the CST process for specific removal of contaminants. The CST process is far less capital intensive than the traditional lime treatment plants. Additionally, our process does not create substantial material handling problems inherent with the creation of vast amounts of gypsum in lime-based systems.

### **CST Service Business Concept**

CST's core business focus is to operate as a turnkey water treatment service company. We do not intend to market our patented equipment as a manufacturer, but rather implement, own and operate the plants as a water treatment service which specifically addresses each client's particular environmental discharge problems. This business strategy will allow CST to protect its technology from potential patent infringement in the near term and provide the framework for long term profitability as project financing is amortized and continued revenues become a profit contribution. This is fundamentally a different approach from that offered by our competition, which generally prescribes a traditional engineering approach requiring construction of capital intensive lime treatment facilities. We have received a positive response to our service business approach from clients who require contaminated water treatment. The concept of a client being able to solve significant discharge problems without a considerable up front capital expenditure is very attractive.

Relationship of Affiliated Companies and Ownership Structure of  
Chemical Separation Technology, Inc.



## TECHNICAL DESCRIPTION

### **Detail Description of Process**

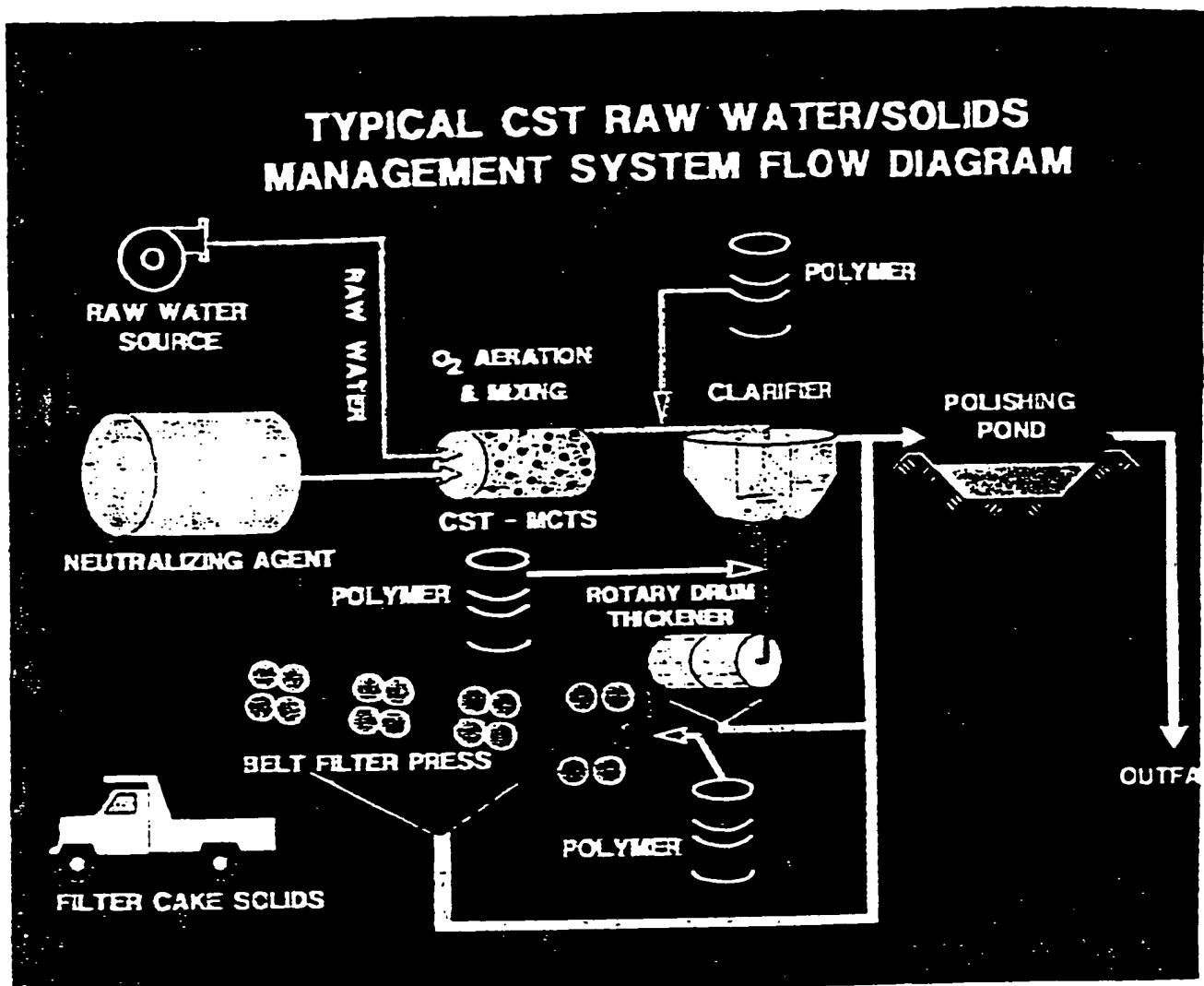
An increasing concern for the quality of the water released to the environment led to the development of the patented CST process and equipment. Acidic and metals-bearing effluents from coal and metals mines and from coal-fired utility power plant coal and flyash deposits will severely degrade streams, rivers or lakes unless remedial actions are taken. The currently proposed Clean Water Act Reauthorization Bills before Congress will mandate corrective actions and potentially increase civil and criminal penalties for non-compliance with the Clean Water Act standards.

The equipment is comprised of a cylindrical reaction vessel with internal baffles, computer-controlled pH monitoring and recording equipment, an aeration motor, piping and chemical storage tanks. Sludge thickening, dewatering and filtration equipment or clarifiers reduces the solid wastes to the minimal volume for efficient disposal. The equipment is easily scaled to handle the volume flowrate of the individual problem, ranging from as small as ten gallons per minute to major flows of 10,000 gallons per minute. Larger flows can be handled with multiple units to the flowrates required. The CST reactor and ancillary equipment is portable, either with skid-mounted or on flatbed trailers, for the flowrates from ten to 500 gallons per minute; permanent equipment can be installed either within buildings or outside, depending upon the individual requirements.

The CST process shown in FIGURE 2 on the following page, receives acidic leachate from coal, flyash or metals mines containing metallic constituents which are fed to the CST reactor where sodium hydroxide is simultaneously and vigorously mixed with air to create localized high concentrations of neutralizing and oxidizing conditions. As a result, the reaction times are markedly reduced, thereby eliminating large settling ponds and level areas which are generally not available, particularly in mountainous areas. Through the use of state-of-the-art computerized process control instrumentation, the pH is held at a constant level allowing the point precipitation of the metals in solution. The CST process is much less capital intensive than the traditional lime treatment plants and does not create substantial waste disposal problems that are inherent with the disposal of vast amounts of un-reacted lime with solids from the lime-based systems.

Acidic leachate of about 230 gallons per minute from an eastern coal-fired power plant flyash disposal site is being treated with a CST facility to provide water effluents meeting EPA standards for release to surface waters and a sludge that is suitable for disposal in the utility's disposal field. TABLE 1 illustrates the results that are routinely achieved. Although the water is not immediately suitable for drinking purposes, it is acceptable for industrial use or discharge to the local watershed. Greater than 99% of the iron and 94% of the manganese are precipitated in less than ten minutes at a routine pH of 7 to 8.

FIGURE 2



**ADVANTAGES OF THE CST SYSTEM**

**Effective and Efficient**

- Rapid Precipitation
- Instantaneous Reaction
- Precise pH Control
- Optimum Use of Chemicals
- Measurable and Documented Results
- Constant System Monitoring and Reporting
- Monitored and Controlled Remotely
- Meets All EPA NPDES Discharge Regulations

**Sensible and Economical**

- Modular System Construction—Quick Installation
- Service Contracts:
  - No Capital Investment
  - No Labor Problems
- Small Site Area Required
- No Multiple, Large Holding Basins
- Efficient Sludge Removal and Disposal

Flocculation, dewatering and belt-filtration recovers about 15,000 pounds of solids per day. The facility is sized so that it can routinely accept and process higher level excursions of acid and metal concentrations up to about 6000 and 7000 mg/liter, respectively. Combining the specific plant process designs with instrumentation controls, provides the CST process with a wide range of flexibility to handle changing flow rates, pH balance, contained metals and acidity levels.

TABLE 1. Western Pennsylvania Water Quality Data for January 28, 1991

	<u>Leachate</u>	<u>Effluent</u>	<u>Percent Removal</u>
Flow, US gpm	230		
pH	2.9	7.1	—
Iron, mg/l	1,687	<1	99.9
Manganese, mg/l	17	<1	94.0
Aluminum, mg/l	114	17	85.0
Acidity, mg CO <sub>3</sub> /l	4,040	0	—
TSS, mg/l	1,870	12	—

Further reduction in the aluminum and other trace metals such as a copper, zinc, lead and magnesium is achievable by controlled pH and oxidation modifications using the CST process. Other treatment steps, including lime-soda ash softening (to meet hardness requirements), removal of organics (by carbon adsorption), turbidity control (by coagulation and filtration), and disinfection (by chlorination), can be added to improve water quality that will potentially comply with drinking water standards.

### Patent Discussion

The CST Patent (United States Patent Number 4,749,497) provides a new method to treat acidic and metals-contaminated leachates from utility plants, coal mines and metals mines which produces treated water that meets EPA Standards for discharge to the environment. The contained metals are precipitated and dewatered to permit discharge to approved disposal sites or for recycle of the contained metals. This unique process develops a continuous stream, mobile treatment system that can economically treat flows from 10 to 1,000 GPM, or when grouped from 1,000 to 10,000 GPM. The combination of reaction geometry and control systems establish the optimum point of mix, neutralization chemical and oxidant addition to achieve a one to one stoichiometric reaction. The Proportional Integral Differential (PID) control system continuously reads the chemical reaction pH, which allows execution of precise, instantaneous control over pH fluctuations in a very narrow band. Radio and telephone telemetry monitors required fail safe points in the process which allows the system to be operated remotely without the need for continuous on-site labor support. Additional down

stream processes have been developed and tested which separate and dewater oxide metals to 15% dry solids. Process patents for this advanced technology are being applied for at the present time. A copy of the patent document is provided in the Appendix section of this document.

### Competitive Processes

Processes which focus on the addition of lime have been the conventional treatment of industrial acidic streams and discharge sources. Addition of lime or lime-sodium carbonate increases the pH and the resultant slurry is mixed to promote neutralization and oxidation. Generally, such treatment facilities are very capital intensive, utilizing complex mixing, lime slaking, and other labor intensive equipment. This process requires large settling basins or ponds to permit the mixing and slow precipitation to occur. Control of pH with lime depends on the extent of solid-liquid mixing and the reactivity of the lime which is a function of particle size, slaking, and the interference of calcium carbonate formed by the absorption of carbon dioxide. Lime processes provide only a high pH range in that a pH near neutral cannot be maintained with the solid-liquid reaction of lime with water. Solids from lime processes include the precipitated metals, but also large quantities of un-reacted lime which requires extensive material handling requirements.

Sodium hydroxide is not generally used to neutralize acid and precipitate metals in a traditional "lime-type" treatment process. This is because it retains most of the disadvantages inherent in the process such as high capital cost and the requirement for large solids settling basins. While the chemistry is not new, other processes using sodium hydroxide have not been commercially demonstrated or perfected like the CST process which is in full commercial operation. Competition from emerging technologies could increase as CST successfully penetrates the marketplace and generally increases its market share.

### Compliance with EPA Discharge Standards

An increasing concern for the quality of the water released to the environment led to the development of the patented CST process and equipment. Acidic and metals-bearing leachates from coal and metals mines and from coal-fired utility power plant coal and flyash deposits will severely degrade streams, rivers or lakes with iron, manganese and acids unless remedial actions are taken. The currently proposed Clean Water Act Reauthorization Bills before Congress will mandate corrective actions and potentially increase civil and criminal penalties for non-compliance with the Clean Water Act Standards.

The CST process fully meets the EPA standards for discharge of industrial water to the environment. Current Pennsylvania discharge standards for iron and manganese are 4 ppm and 2 ppm, respectively with a pH range of 6 to 8. The CST treated water contains less than 1 ppm of either iron or manganese and has a pH range of 7 to 7.5. Other states are developing discharge standards which are expected to be similar to Pennsylvania for industrial water and follow EPA standards for drinking water.

### Significant Provisions Clean Water Act

Senate Bill 1081 and House Bills 2029, 1330, 251, 404, and 2400, if authorized, would result in more restrictive effluent limitations and will require more extensive pollution prevention efforts. Key aspects of these bills would:

- o Require applications of the "Best Available Control Technology (BACT or BAT) to all pollutants.
- o Require sources to utilize techniques for pollution prevention and waste reduction.
- o Prohibit or limit the release of pollutants to environmental media, including ground water, to the extent such is economically and technologically feasible.
- o Require states to adopt administrative civil penalty provisions similar to the EPA under the Clean Water Act and increase civil and criminal penalties for noncompliance. Mandatory environmental audits would be required prior to permit renewals.

# United States Patent [19]

Inglis

[11] Patent Number: 4,652,381

[45] Date of Patent: Mar. 24, 1987

[54] BATTERY PLANT WASTE WATER TREATMENT PROCESS

[75] Inventor: James L. Inglis, Liberty, Mo.

[73] Assignee: Farmland Industries, Inc., Kansas City, Mo.

[21] Appl. No.: 757,351

[22] Filed: Jul. 22, 1983

[51] Int. Cl. <sup>4</sup> C02F 1/52

[52] U.S. Cl. 210/726; 210/912; 423/35; 423/92

[58] Field of Search 210/712, 713, 721, 722, 210/724, 726, 727, 912; 423/92, 35

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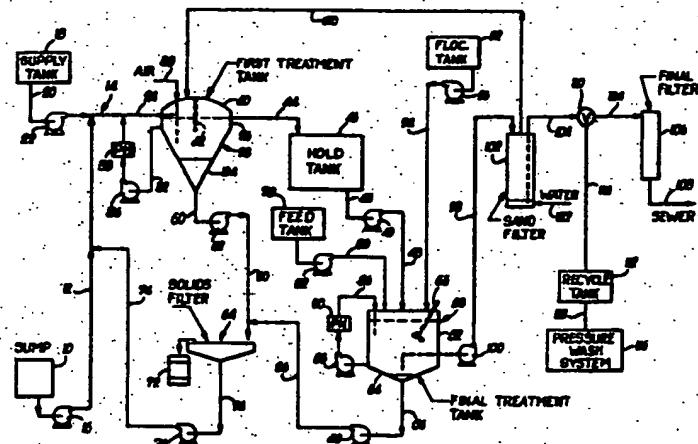
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Primary Examiner—Peter Hruskoci  
Attorney, Agent, or Firm—Schmidt, Johnson, Hovey, & Williams

[57] ABSTRACT

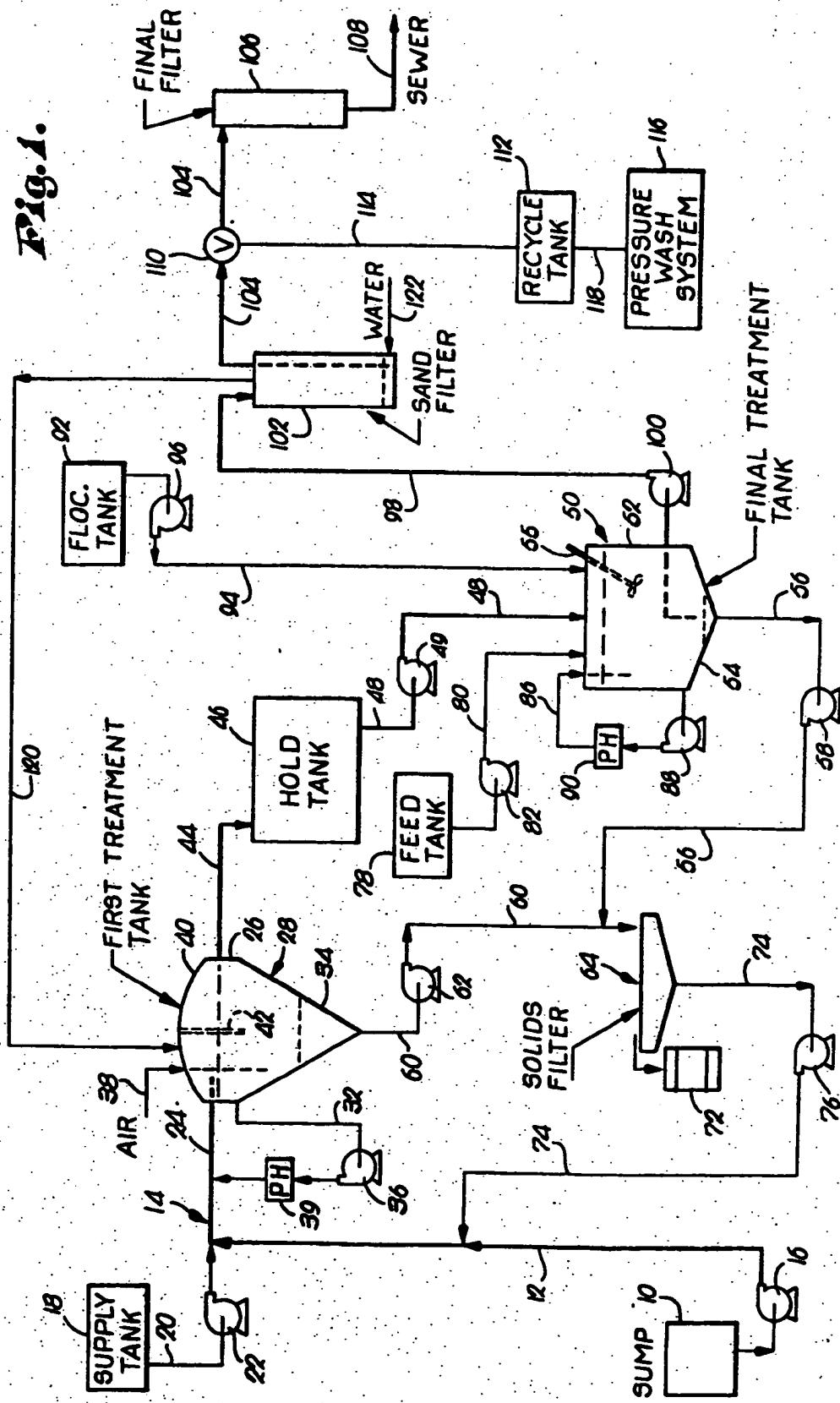
A process of treating industrial waste water contaminated with environmentally unacceptable amounts of sulfuric acid and heavy metals such as lead, copper or zinc is disclosed which permits lowering of the concentration of the contaminants to a level permitting discharge to the sewer. Waste water resulting from floor wash and spray washing of lead acid batteries prior to shipment from the manufacturing facility contains sufficient sulfuric acid to cause the pH to normally be at a level of about 2 along with heavy metal contaminants present in concentrations which require treatment for removal before the discharge water will meet EPA standards. The water to be treated is directed to a first reaction and settling vessel where calcium carbonate is added along with an oxidation medium such as air which also functions to stir the stored waste water. Sufficient calcium carbonate is added to bring the pH of the solution to a level of about 5 and at the same time react with the heavy metals present such as lead, copper or zinc. Calcium sulfate and respective heavy metal carbonates precipitate and settle to the bottom of the treatment zone where they may be readily removed. In a second treatment vessel, calcium hydroxide along with enough calcium carbonate to maintain an excess of carbonate ion are added to complete separation of the heavy metals. Final removal of precipitate from the solution is accomplished through a suitable filter.

3 Claims, 7 Drawing Figures



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Fig. 1.



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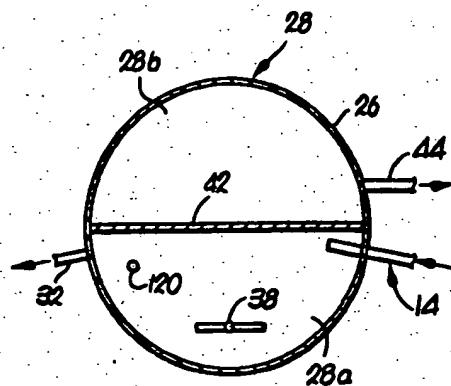


Fig. 3.

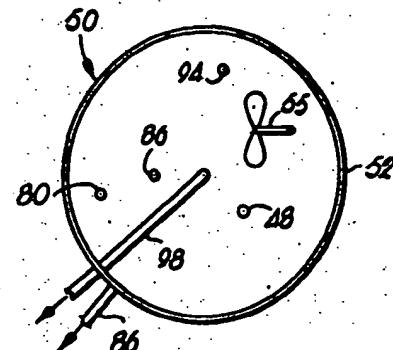


Fig. 5.

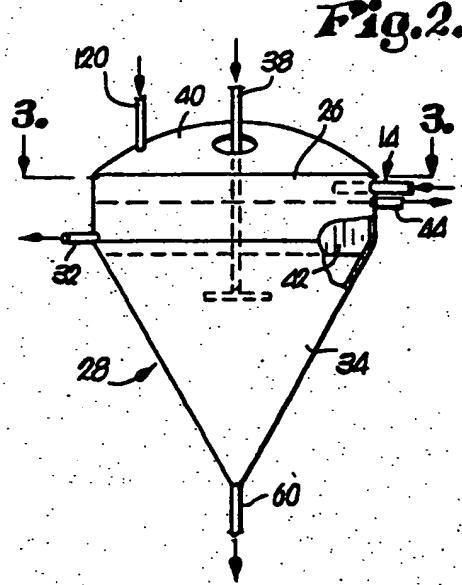


Fig. 2.

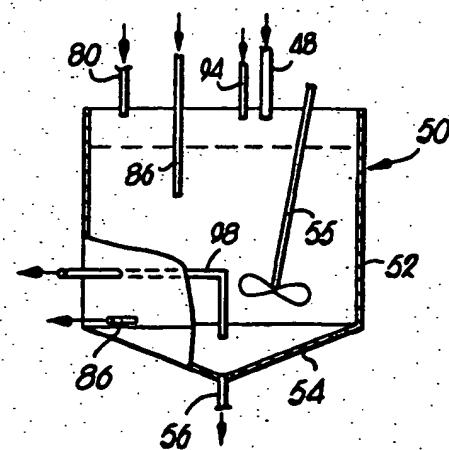


Fig. 4.

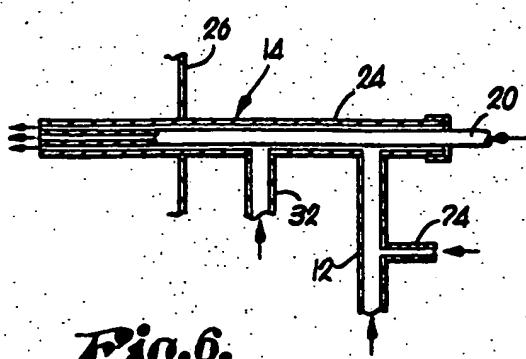


Fig. 6.

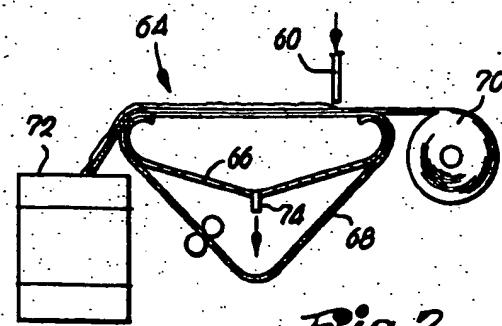


Fig. 7.

## BATTERY PLANT WASTE WATER TREATMENT PROCESS

### BACKGROUND OF THE INVENTION

#### 1. Field of the Invention

This invention relates to a process for treating industrial waste water containing contaminants in the nature of sulfuric acid and heavy metals such as lead, copper and zinc, to lower the concentration of such contaminants to a level permitting discharge of the waste water to the sewer.

The process has particular utility in the treatment of waste water resulting from the washing of lead acid batteries and plant floors causing the water to contain residual quantities of sulfuric acid and metal constituents primarily made up of lead and copper.

Use of the process permits recycling of treated water back to the plant washing process thus lowering the overall operating costs of the battery plant. Water which is periodically discharged to the sewer has acid and metal levels far below those permitted in governmental regulations.

#### 2. Description of the Prior Art

The assignee of this application has long operated a lead acid battery plant. During the manufacture of lead acid storage batteries, it has been conventional for some time to suspend the batteries from a conveyor line for passage through a water spray cleaning system. Water is sprayed onto the batteries to thoroughly clean the exterior surface of each battery. The spent water is collected through floor drains and directed to a storage area. Following washing of the battery cases, the batteries are generally dried with pressurized air. A large amount of waste water also comes from washing down the floors in the plant.

The waste water from the lead acid battery wash and plant floor washings cannot be reused in the manufacturing process in part because iron in the waste water is deleterious to battery life, and the high acidity and lead content preclude discharge direct to a sewer without subjecting the water to a treatment operation. In the past, the assignee hereof has employed a mixture of sodium hydroxide and sodium sulfide to remove lead from the waste water. Some recycling of the contaminated water was carried out, but build up of a sodium sulfate salt prevented effective use of the treatment on an ongoing basis. EPA regulations provide that only 0.15 lb. of lead and 0.76 lb. of copper may be found in the waste water per million pounds of lead used per month in order to permit discharge of the waste water into the sewer.

It has been suggested in "Development Document for Effluent Limitation Guidelines and Standards for the Battery Manufacturing Point Source Category", October 1982, EPA 440/1-82/067-b, U.S. Environmental Protection Agency, that sodium hydroxide be added to the waste water to be treated and the resulting  $Pb(OH)_2$  allowed to settle for removal.  $Pb(OH)_2$  has a minimum solubility point on its solubility curve at pH 9.5. Thus, the discharge to sewer should be 9 to 10 and never below 8. Below pH 8, the solubility of  $Pb(OH)_2$  is such that meeting the discharge requirements would be very difficult.

The development document also describes a method of using soda ash ( $Na_2CO_3$ ) and caustic soda ( $NaOH$ ) to treat the waste when no recycling is required to give

low lead concentration. In this procedure, the lead is precipitated as the carbonate at a pH above 7.

Hamala, et al. in Volume 11, Issue No. 1 at pages 243, et seq. (1977) of Water Research ("Calcium Carbonate in the Removal of Iron and Lead From Dilute Waste Water") indicate that calcium carbonate has been used in the treatment of acid mine waters and steel pickle liquors at pH levels of 8 or more. However, precipitation of ferric hydroxide is believed to interfere with settling of calcium sulfate and lead carbonate. The Hamala, et al. process does not provide a treated waste water which will satisfy current EPA sewer discharge regulations.

Hoak, et al. in Volume 37, Issue No. 6, Industrial and Engineering Chemistry, June 1945 at pages 553, et seq. ("Treatment of Spent Pickling Liquors with Limestone and Lime") describe a process of treating spent pickling liquor with limestone and lime. Quick lime ( $CaO$ ) was used to elevate the pH to the alkaline side before air was added to achieve rapid oxidation.

### SUMMARY OF THE INVENTION

Waste water resulting from washing the exterior surfaces of lead acid batteries and from washing the plant floor is stored and when a suitable quantity thereof is collected, it is directed into a combination reaction and settling vessel wherein calcium carbonate is metered into the waste water to maintain a pH of about 5. Air is introduced into the waste water in the reaction vessel for the dual purpose of agitating the solution and assuring intimate admixture of the calcium carbonate and water solution, and to oxidize iron present in the waste water to ferric oxide precipitate. Calcium sulfate and lead, copper, zinc and other heavy metal carbonates thus formed settle out upon standing of the solution and may be removed from the treatment vessel.

The liquid is directed to a suitable hold tank and then introduced into a final treatment vessel where the solution is mechanically agitated and a slurry of about 12% by weight of mixed limes (20% calcium carbonate and 80% calcium hydroxide) is added. Again, precipitates are allowed to settle out with the clarified solution being directed overhead and then passed through a sand filter to effect final removal of a remaining solids from the water, which is then in condition for recycling to the plant wash water system.

### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic diagram of a preferred process for carrying out the invention hereof and illustrates in diagrammatic form various pieces of equipment which may be advantageously employed to practice the process;

FIG. 2 is an enlarged fragmentary view of the first treatment tank on a somewhat enlarged scale illustrating the construction of a preferred vessel and the manner in which liquid and gas are caused to inflow and liquid is allowed to outflow therefrom;

FIG. 3 is a horizontal cross-sectional view taken substantially along the line 3-3 of FIG. 2;

FIG. 4 is an enlarged fragmentary view of the final treatment tank again illustrating the construction of a preferred vessel and the manner in which liquid is caused to inflow and outflow therefrom;

FIG. 5 is a horizontal view looking downwardly on the open top vessel of FIG. 4;

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FIG. 6 is a depiction of suitable flow direction piping for the liquid streams introduced into the vessel of FIG. 2; and

FIG. 7 is a schematic illustration of the solids filter used in association with the final treatment tank of FIG. 4.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

In a typical lead acid battery plant which has a total lead input of approximately 1,000,000 pounds per month, current EPA regulations limit the waste water discharge to sewer contaminants to 0.15 lb. of lead and 0.76 lb. copper per month. If 50,000 lbs. of lead per day is consumed, the daily limit of lead and copper in waste water to sewer would be no more than 0.016 lb. of lead and 0.072 lb. of copper. Current EPA regulations are generally based on the total amount of contaminants discharged over a selected period of time and not their concentration in the waste stream. Compliance is determined by measuring the concentrations of the metals in the waste stream and multiplying it by the waste stream volume. New regulations thereby encourage the reduction of waste stream volumes. With a lead concentration of 1.28 ppm in the waste stream, only 1,200 gallons of waste water could be discharged in a 24 hour period. However, at 0.2 ppm of lead in the waste water, 7,600 gallons of treated waste water could be discharged to the sewer in a 24 hour period.

The process of this invention produces a waste water discharge to sewer containing less than 0.2 ppm of lead and an even lesser amount of copper. This compares with prior processes which gave a soluble lead concentration in the waste water above 10 ppm and to a certain extent were erratic and unpredictable.

Metal contaminants are precipitated as carbonate and carbonate-hydroxide basic salts. The solids are separated by filtration and sent to a landfill. It is a feature of this process that precipitation of these salts is affected at a pH value lower than those indicated to be feasible in the prior art. Precipitation of lead, copper and zinc carbonate salts have been found to occur at a pH as low as 4.5 to 5.0. The carbonates thus precipitated at a pH below 7 are less soluble than lead, copper and zinc hydroxide which are precipitated at a pH above 7. As a result, the removal of lead, copper and zinc salts is more efficient using a carbonate system than is the case when reliance is placed on precipitation of the metals as hydroxides above pH 7, as in the processes of the prior art and particularly, the EPA referenced methods. It is also a feature of this process that trace amounts of iron are removed by sparging air into the treatment vessel to precipitate the iron as  $Fe_2O_3$ . Precipitation of the iron by oxidation with air may readily be accomplished without adversely affecting removal of lead, copper and zinc as carbonates in an acidic medium. Furthermore, removal of the iron as ferric oxide at this point in the process avoids formation of a gelatinous, difficulty filterable iron precipitate which takes place above pH 7 and that would clog a filter used to remove the precipitates.

Using a 1,000,000 pound per month lead utilization lead acid battery plant as a exemplary standard, the present method treats 1,200 gallons a day of waste water from the battery wash operation to be processed daily with 200 gallons being discharged to the sewer and 1,000 gallons being recycled for reuse.

Waste water collected from the lead acid battery plant wash lines is stored in sump 10 which has an outlet line, 12 leading to common supply line 14. Pump 16 is interposed in line 12 adjacent sump 10. A calcium carbonate slurry tank 18 is joined to supply line 14 through the provision of a line 20 having a pump 22 thereina.

As is best seen in FIG. 6, supply line 14 includes an outer pipe 24 which extends through the cylindrical wall section 26 of generally conical first treatment tank 28. Line 12 is coupled directly to pipe 24 while pipe 20 extends into the interior of pipe 24 and is coaxial with the latter in spaced relationship from the interior wall of the latter to define a cylindrical passage around the periphery of pipe 20. Line 20 terminates within the interior of treatment tank 28 at the innermost extremity of pipe 24.

A recycle line 32 connected to first treatment tank 28 at the zone of merger of cylindrical section 26 with the conical segment 34 of such tank has a pump 36 therein and leads to pipe 24 for communication with the latter between pipe 12 and the innermost end of pipe 24. A pH controller 38 in line 32 is connected with pump 22 to control delivery of calcium carbonate from supply tank 18 to first treatment tank 28. Line 38 extending downwardly into the spherical top section 40 of treatment tank 28 serves to introduce an oxidizing medium such as air into the body of liquid contained in tank 28. The lowermost end of air line 38 terminates in a tee having a series of openings in the horizontal portioa thereof for delivery of air in bubble form into the interior of the tank 28. An upright baffle 42 (see FIGS. 1-3) within tank 28 and spanning cylindrical section 26 in bisecting relationship thereto is joined to the underside of spherical top section 40 and projects downwardly into the tank a distance somewhat below the lower circular line defined by the bottom of cylindrical section 26. Air line 38 extends into tank 28 through an open manhole on the same side of baffle 42 as supply line 14 (see FIGS. 2 and 3).

Line 44 joined to cylindrical section 26 of first treatment tank 28 and communicating with the interior thereof, leads to a holding tank 46. A feed line 48 joins the lower end of holding tank 46 with an open top final treatment tank broadly designated 50. A delivery pump 49 is interposed in feed line 48. The cylindrical sidewall 52 of tank 50 terminates at the lower peripheral edge thereof in a shallow coned bottom 54. Mechanical agitator 53 is provided in tank 50 for stirring the contents thereof. Line 56 communicating with the final treatment tank 50 at the bottom of conical section 54 and provided with a pump 58 therein leads to a solids removal line 60 connected to the lower conical section 34 of treatment tank 28. Line 60 has a pump 62 therein and terminates in direct overlying relationship to a solids filter broadly designated 64.

As depicted schematically in FIG. 7, the solids filter 64 includes an underlying angle bottom liquid collector 66 which receives an endless belt conveyor 68 which supports a disposable filter paper 70. Thus, solids deposited onto the filter paper supported by conveyor 68 are directed into the container 72 while liquid draining through the filter paper is returned to outlet line 12 via line 74 having a pump 76 therein.

Feed tank 78 for a slurry of calcium carbonate and calcium hydroxide has a delivery line 80 joined to the lower end thereof which feeds into the open upper end of final treatment tank 50. Pump 82 is interposed in line 80. Recycle line 86 extending from the tank 50 at the

lower periphery of the cylindrical section 52 thereof loops back over the top of tank 50 and delivers recycle liquid into the open end thereof. A pump 88 is provided in line 86 while pH controller 90 is operably connected to pump 82 for controlling delivery of slurry from feed tank 78 into tank 50. Flocculant storage tank 92 has a delivery pipe 94 joined thereto which extends to the open top of tank 50 and is provided with a feed pump 96 therein.

An outlet line 98 having a pump 100 extends from the lower part of final treatment tank 50 to the inlet of an upright sand filter 102. An outlet line 104 extending from the lower end of filter 102 is joined to the inlet of a final filter 106 which has an outlet pipe 108 leading to the sewer. Valve 110 in line 104 permits selective direction of waste water into recycle storage tank 112 via line 114. Recycle tank 112 is in turn connected to the pressure wash system 116 via line 118. Filter purge line 120 joins the sand filter 102 to the top spherical section 40 of first treatment tank 28. Backwash water is supplied to the lower end of sand filter 102 by a fresh water supply line 122.

#### Operation of Preferred Embodiment

During the manufacture of lead acid batteries, it is conventional to wash the batteries prior to packaging. 25 Generally speaking, the batteries are supported by a conveyor which transports the units through a spray wash where they are subjected to three separate high-pressure spray washes. The wash water removes residual lead and sulfuric acid whereupon the batteries are 30 blown dry. Waste water collected from the washing operation is directed via floor drains to the sump 10 along with the water used to wash the plant floor for treatment to permit recycling thereof to the wash water systems, with a certain proportion allowed to flow into 35 a sewer line. The waste water is intermittently pumped via line 12 into the first treatment tank 28 through supply line 14. The waste water from pump 18 is generally saturated with lead sulfate and often contains suspended 40  $PbSO_4$ . Total lead in the waste water can run as high was 500 ppm. In addition, the waste water collected in sump 10 typically contains about 1%  $H_2SO_4$  giving the water a pH below 2. Iron is normally present at levels about 50 ppm while copper can be as high as 10 ppm. For a plant consuming about 1,000,000 lbs. of lead per 45 month, the exemplary lead acid battery wash and floor wash systems will generate about 1,200 to 1,300 gallons per day of waste water collected in sump 10. The temperature of such water will be approximately the ambient temperature of the plant.

For a typical wash water system as described, treatment tank 28 should have a working capacity of approximately 1,300 gallons. Treatment of waste water in the tank 28 can be accomplished on a continuous or batch basis. Waste water collected in sump 10 is pumped to vessel 28 via line 12 until the treatment tank is substantially filled with the waste water to be treated. Thereafter, delivery of water to the treatment tank may be on an intermittent basis or substantially continuously, if the flow is controlled such that there is adequate residence time of the waste water in tank 28 to assure optimum precipitation of calcium sulfate and heavy metal carbonates which settle out of the solution and are removed from the tank via line 60. In order to assure collection of solids at the bottom of the tank 28, inclination of the conical wall 34 at an angle of about 30° has been found to produce satisfactory results. The divider baffle 42 desirably is of a depth such that it is connected

10 to and extends from the spherical top 40 of the tank to a horizontal line about 4 inches below the circular, circumferentially extending lower margin of the cylindrical section 26 of tank 28, thus dividing the upper part 15 of the treatment tank 28 into a treatment section 28a (i.e. the upper portion of the tank 28 into which the waste water is directed via line 14) and a settling section 28b (i.e. the upper portion of the tank 28 from which the liquid is allowed to egress from the tank through line 44; see FIG. 3).

10 Air is sparged continuously into the interior of treatment tank 28 through line 38 to effect some mixing of the liquid contained in the tank and to also provide an adequate supply of an oxidizing agent (in this case  $O_2$ ) to convert the iron contained in the waste water to ferric oxide ( $Fe_2O_3$ ). However, the major mixing of liquid in treatment tank 28 is provided by recirculation of liquid through line 32 by pump 36.

15 Calcium carbonate and recycled waste water are mixed in supply tank 18 and the slurry thus produced is pumped via line 28 into the interior of treatment tank 28 extending coaxially through pipe 24. Operation of pump 22 controlling delivery of calcium carbonate slurry from supply tank 18 to first treatment tank 28 is under the control of pH metering unit 39. Although the specific concentration of the calcium carbonate in supply tank 18 is not highly critical, a 12% by weight slurry of the calcium carbonate may readily be pumped into the 20 interior of the vessel 28. A normal rate during operation of pump 22 has been found to be about 80 pounds of calcium carbonate per 1,000 gallons of waste water. The amount required may vary within relatively large limits and depends in large measure on how well sulfuric acid spills are controlled in the lead acid battery 25 manufacturing facility. The main consideration is that the amount of calcium carbonate furnished to first treatment tank 28 and the period of delivery thereof to the treatment vessel be controlled such that the pH of the waste water contained in treatment tank 28 be maintained at a pH of from about 4.8 to 5. Thus, controller 39 should be set up such that it initiates operation of pump 22 to deliver calcium carbonate slurry to the interior of first treatment tank 28 when the pH of the recycle water flowing through pipe 32 reaches a level of about 4.8, and then functions to discontinue energization of pump 22 when the pH of the solution in the recycle line 32 rises to a level of about 5.0. The calcium carbonate reacts with sulfuric acid in the waste water to produce calcium sulfate which precipitates from the solution as  $CaSO_4 \cdot 2H_2O$ . In addition, heavy metals present in the waste water such as lead, copper and zinc react with the calcium carbonate to form carbonate precipitates which, along with the calcium sulfate settle to the bottom of the tank 28 for removal via line 60. The majority of the carbonate however reacts with the sulfuric acid and is expelled as  $CO_2$  gas.

20 From one to three times per day solids which have collected in the lower conical section 34 of tank 28 are removed via line 60 during operation of pump 62. The high solids content liquid delivered from line 60 is deposited on the filter paper 78 of filter 64 where such solids are directed by conveyor 68 into the container 72. For most efficient operation of the filter 64, the unit is 25 preferably of the type causing a vacuum to be imposed on the conveyor belt 68 with removed water being directed via line 74 back to supply line 12 through the action of pump 76. Solids collected in container 72

(which for example may be a 55 gallon drum) are shipped to a landfill.

By virtue of the fact that the waste water in first treatment tank 28 is not only recirculated but also must flow beneath the lower edge of baffle 42 from reaction zone 28a to the quiescent zone 28b, there is efficient interaction between the calcium carbonate and the sulfuric acid and heavy metals contained in the waste water. Liquid overflow from treatment tank 28 leaves the latter via line 44 and is directed into the hold tank 46 by gravity flow. The pH of the waste water leaving vessel 28 is in the order of 5 to 7 because the calcium carbonate continues to react in the static section 28b of treatment tank 28. The soluble lead, copper and zinc in the waste water leaving first treatment tank 28 have been found to be less than 0.2 ppm but the totals may be above 10 ppm because of fine particles that are entrained in the liquid. On the other hand, the soluble iron content has been determined to be less than about 0.5 ppm if oxidation has been properly carried out in the reaction zone 28a of tank 28. Desirably, hold tank 46 is of a size to hold about 1,500 gallons of solution where tank 28 has a capacity of 1,300 gallons (although an emergency overflow hold tank of an additional 1,500 gallons capacity may be provided in association with hold tank 46 if additional hold capacity is desired).

Preferably, the contents of the hold tank 46 are pumped once a day into the final treatment tank 50 via line 48. An approximate 12% by weight solids slurry of a mixture of 20%  $\text{CaCO}_3$  and 80%  $\text{Ca}(\text{OH})_2$  (providing a mole ratio of carbonate to hydroxide of about 1:10) is mixed in feed tank 78 and pumped into final treatment tank 50 through line 80 under the control of pump 82. Again the 12% concentration of the calcium carbonate-calcium hydroxide mixture may readily be pumped through line 80 to the tank 50. The ratio of  $\text{CaCO}_3$  to  $\text{Ca}(\text{OH})_2$  is such as to provide an excess of carbonate ions in final treatment tank 50. Calcium hydroxide is necessary to raise the pH to the desired level in the final treatment. Under normal operating conditions there is little, if any, reaction of calcium carbonate in the treatment tank 50. The solubility of lead hydroxide (2.1 ppm as lead) is too high to meet specifications; thus, steps are taken to preclude formation of lead hydroxide in the process. Typical usage of  $\text{Ca}(\text{OH})_2$  is no more than about 8 pounds per 1,000 gallons of waste water.

Mixing of the solution in treatment tank 50 is assured by a slow speed agitator in the nature of a propeller mixer 55. In addition, the solution in treatment tank 50 is recirculated through line 86 under control of pump 88. The pH controller 90 functionally coupled to pump 82 controls the operation of the latter to feed the  $\text{CaCO}_3$ - $\text{Ca}(\text{OH})_2$  slurry into treatment tank 50 upon demand. The controller is adjusted between a pH of 8.0 and 9.0. When the pH of the solution reaches 9.0, operation of pump 82 is discontinued, whereupon the contents of treatment tank 50 are mixed for at least about 1 hour and then an anionic polyelectrolyte flocculant contained in tank 92 is pumped via line 94 into the final treatment tank 50. A preferred flocculant giving desirable results has been found to be Mitifloc 51 supplied by Mitco Incorporated, Grand Rapids, Mich. After approximately 1 minute of mixing, the system is shut off and the contents of final treatment tank 50 are allowed to settle for from 2 to 24 hours and most preferably about 20 hours.

Heavy metal carbonates and any residual calcium sulfate collected in the bottom of final treatment tank 50

are returned to line 68 via line 56 under the control of pump 58 for filtering of such solids in association with those received from first treatment tank 28. The liquid is thereby returned to first treatment tank 28 through line 74, line 12 and supply line 34.

The clear supernatant resulting from quiescent settling of solids from the waste water in tank 50 is pumped via transfer line 98 under the control of pump 100 to sand filter 102 to remove suspended particles in the waste water. Backwashing of the sand filter 102 should be carried out about once a week with water being supplied for this purpose through line 22 and the resulting solids removed being directed back into first treatment tank 28 through line 128.

The discharge from sand filter 102 is directed via line 104 to a three way valve 110 with one line 104 therefrom going to the final filter 106 while another line 114 returns water to the recycle tank 112 which is connected to the pressure wash system 116 through line 118. The waste water treated each day is divided such that about an average of 80% thereof is returned to the pressure wash system 116 while the remaining 20% goes to the final filter 106 for ultimate discharge to the sewer via line 108. The treated waste water leaving final filter 106 is normally well within regulations established by the EPA. However, final filter 106 desirably containing a 5 micron bag filter is a safety measure if a malfunction of sand filter 102 occurs for some reason, or if some particularly fine material is not removed by settling in the final treatment tank and passed through filter 102. In a preferred arrangement, recycle tank 112 is of dimensions holding approximately 4 days of water for supply to the plant pressure wash system 116.

Oxidation of iron contained in the waste water while the latter is held in tank 28 prevents formation of a hard to filter iron precipitate in the final treatment tank 50. If unoxidized iron is allowed to reach tank 50, filtration problems can occur above pH 7 (noting in this respect that the pH of the waste water in tank 50 is desirably at least about 8 up to about 9),  $\text{Fe}(\text{OH})_3$  which forms can be very difficult to filter. Air sparging of the waste water in the first treatment tank 28 not only permits utilization of the air as an agitation medium, but also assures conversion of the iron to  $\text{Fe}_2\text{O}_3$  which precipitates and may be readily filtered out in the filter 64.

A particularly important feature of the present process is the fact that a relatively inexpensive commodity of commerce (limestone, i.e. calcium carbonate) may be employed to effect removal of sulfuric acid and heavy metals from the battery acid plant waste water. Limestone not only may be obtained at a reasonable cost but is available in a particle size that assures efficient reaction thereof with the acid and heavy metals in the waste water. Typically, calcium carbonate sold as paint pigment or an animal feed additive has a particle size of approximately 5 microns. Desirably, the calcium carbonate is of a particle size such that at least about 90% will pass through a 100 mesh screen.

In spite of the low solubility of  $\text{CaCO}_3$  in water, the present process takes advantage of the fact that calcium carbonate becomes more soluble in a solution saturated with  $\text{CO}_2$ . Since the reaction of  $\text{H}_2\text{SO}_4$  with  $\text{CaCO}_3$  results in release of  $\text{CO}_2$  thus saturating with the solution of carbon dioxide, the carbonate ion is present in sufficient supply to assure precipitation of lead and other heavy metals as carbonates. It was therefore unexpectedly found, contrary to the teachings of the prior art as for example, indicated in the above-referenced

EPA Development Document, that effective removal of lead and other heavy metals may be accomplished at a pH in the order of 5.

Although best results are obtained at the lead cost using limestone ( $\text{CaCO}_3$ ) as the precipitating agent for the sulfuric acid and heavy metals in the waste water, other alkali metal and alkaline earth carbonates may be employed. Suitable reactants in this respect would include lithium carbonate, sodium carbonate, potassium carbonate, magnesium carbonate, and barium carbonate.

In the second treatment tank 50, wherein calcium hydroxide in association with calcium carbonate is added to the waste water,  $\text{Ca}(\text{OH})_2$  is the additive of choice for cost reasons but again other alkali metal and alkaline earth hydroxides would be usable such as lithium hydroxide, sodium hydroxide, potassium hydroxide, and barium hydroxide. Use of salts that form soluble metal sulfates will cause a buildup of that metal salt in the water thus precluding recycling of the treated waste water even though lead, copper and zinc are removed to desirable sewer discharge levels.

Results obtained from practicing the process of this invention on an actual plant basis may be summarized as follows (21 days actual plant operation):

Day	Lot No.	Gals.	Copper			Lead		
			To Sewer	pH	PPM	Lbs. To Sewer	PPM	Lbs. To Sewer
1	1003		8.37	0.10		0.12		
2	101R		7.89	0.16		<0.10		
3	102R		8.07	0.13		0.14		
4	103S	130	8.12	0.17	0.00018	0.10	0.00011	
5	105R		9.16	0.21		0.37		
6	106S	910	10.62	0.07	0.00053	<0.10	0.00076	
7	107R		9.02	0.05		<0.10		
8	108R		7.25	0.12		<0.10		
9	109S	270	9.38	0.10	0.00023	<0.10	0.00023	
10	110S	210	7.30	0.16	0.00028	<0.10	0.00018	
11	111R		9.44	0.14		<0.10		
12	112S	730	6.51	0.12	0.00073	0.19	0.00116	
13	113R		6.93	0.06		0.14		
14	114R		6.98	0.05		<0.10		
15	115R		7.11	0.08		0.19		
16	116R		7.05	0.02		<0.10		
17	117S	240	8.43	0.02	0.00004	<0.10	0.00020	
18	118S	310	7.13	0.03	0.00008	<0.10	0.00026	
19	119S	30	7.31	0.03	0.00001	<0.10	0.00003	
20	120S	50	8.01	0.03	0.00002	<0.10	0.00007	
<b>TOTAL</b>		<b>2910</b>			<b>0.00210</b>		<b>0.00293</b>	

This Period:

Lead Consumed: 725.384 lbs.

Days Production: 21

EPA Allowable Discharge:

10  
-continued

5	Copper	Lead
	0.06974 lbs/day 0.55129 lbs/period	0.01105 lbs/day 0.10881 lbs/period
10	EPA Discharge Limits (Lbs. Per Million Lbs. of Lead Consumed):	
	Copper	Lead
10	Daily	1.44
	Monthly	0.36
10		0.32
		0.15

I claim:

1. In a method of treating industrial waste water from a lead acid battery manufacturing plant contaminated with environmentally unacceptable amounts of sulfuric acid along with lead and copper to lower the concentration thereof to levels permitting discharge of the water to a sewer, the improved steps of:

adding an amount of an alkaline earth carbonate selected from the group consisting of calcium, magnesium and barium carbonate and mixtures thereof to the waste water to initially raise the pH thereof to a level which is especially conducive to precipitation of lead and copper carbonate and formation of an alkaline earth sulfate from the sulfuric acid, the amount of alkaline earth carbonate added to the waste water being sufficient to raise the pH of the waste water to a level of about 4 to about 5 to effect formation of an adequate quantity of alkaline earth sulfate and precipitation of lead and copper carbonate such that upon removal of the precipitates, waste water may be discharged to the sewer which meets environmental restrictions imposed on the discharge;

a mixture of alkaline earth carbonate and alkaline earth hydroxide is added during the second addition and removing lead and copper carbonate precipitates from the waste water prior to discharging the latter to the sewer.

2. In a method of treating industrial waste water as set forth in claim 1, wherein adjusting the pH of the waste water following the addition of the alkaline earth carbonate thereto, to a level above about 7 by addition of an alkaline earth hydroxide before effecting removal of precipitates from the waste water thereby preventing the redissolution of suspended lead and copper carbonates; and the mole ratio of carbonate to hydroxide in the second addition to the waste water is about 1 to 10.

3. In a method of treating industrial waste water as set forth in claim 1, wherein the step of adjusting the pH of the waste water to a value above about 7 with an alkaline earth hydroxide is carried out in a manner to cause the waste water to have a pH of about 8 to 9 prior to discharge of the waste water to the sewer.

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CERTIFICATE OF SERVICE

Pursuant to 37 C.F.R. § 1.248 I declare that a true copy of the foregoing "PETITION TO INSTITUTE PUBLIC USE PROCEEDING PURSUANT TO 37 C.F.R. § 1.292" was served by first-class mail, postage prepaid, this 31st day of July 2001 to:

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